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LEAK DETECTION IN M55 DETONATORS BY HELIUM LEAK RATE DETERMINATIONS

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Weight gain due to moisture was measured for simulated M55 detonators with precision drilled holes. The increase varied linearly with the hole diameter. Live detonators with 2- and 5-mil holes, immersed in water for 48 hours, demonstrated decreased sensitivity in functioning tests. The logarithm of leak rates did not vary linearly with wait time in accordance with the leak rate equation, but was found to vary linearly with the logarithm of time.

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The article, "Helium Leak Testing: A New Analysis," by W. E. Briggs and S. G. Burnett is reprinted, with kind permission, from Electronic Packaging and Production, June 1969, copyright © 1969 by Milton S. Kiver Publications, Inc.

INTRODUCTION

The plant modernization program for M55 detonator production will result in high volume output. A number of functions, such as cup inspection, loaded detonator inspection, and sealing, are being improved to meet the output requirement. One of the time consuming tests required for this detonator is moistureproofness (ref 1) wherein samples from detonator lots are immersed in 2 to 3 inches of water, maintained at 15.5°C (60°F) for 48 hours, and then tested for functioning.

Exclusion of moisture within sealed items is essential for safety and proper functioning. In the present case, moisture can affect the sensitivity of the explosive components and, therefore, must be prevented from entering the loaded container after the closure is sealed with lacquer. It is assumed that the proper functioning of the detonators after water immersion indicates either an adequate seal or that the water leakage is below a quantity which would affect the sensitivity of the detonator. However, the moistureproofness test does not quantitatively relate the amount of moisture to the decrease in sensitivity, nor is the test procedure representative of a realistic situation to be encountered by this detonator. The test conditions appear arbitrary without definitive intent. Furthermore, since this is a destructive test, it is felt that a well reasoned scientific basis could define and establish accept/reject criteria for moisture penetration by means of a nondestructive method.

M55 Detonator

The configuration for the M55 detonator is shown in figure 1. The container is an aluminum cup. Prior to being loaded, cups are visually inspected for cuts, holes, punctures, and other likely defects. They are then loaded with three separate increments of explosives. After an aluminum disc is positioned on the last increment, the edge of the cup is symmetrically folded over (crimped) to enclose the contents. These loaded detonators are then manually cleaned and visually inspected for defects, and the closure is sealed with a thin lacquer coating which prevents material from exiting and from penetrating into the aluminum cup at the last obvious opening. Samples from each lot are then tested for moistureproofness and the lot is accepted or rejected according to the applicable requirement.

It was thought that a test method used for fuzes (MIL-STD-331A, Test 118) (ref 2) could also be applied to this item for leak detection by means of halogen and helium gases. Since detonators are sealed without being filled with test gases, and are not provided with a filling port, the "back-pressuring" method (ref 3) must be used. In this method, the item is evacuated to a vacuum of 50-mm Hg and pressurized with the appropriate gas at 15 psia. The leak rate is measured with a mass spectrometer.

The helium leak method was chosen to determine the leak characteristics of M55 detonators so that the feasibility of using this method as a substitute for the moisture-resistance test could be evaluated. Although defects through which moisture can enter the metal cups of the loaded detonators consist of cuts,

tears, and punctures having dimensions not readily measurable, the experimental data were obtained from inert detonators with precisely drilled holes. Inert detonators were used for obvious safety considerations. Hole sizes were 0.0025 cm (0.001 in.), 0.005 cm (0.002 in.), 0.013 cm (0.005 in.), 0.025 cm (0.010 in.), 0.038 cm (0.015 in.), and 0.057 cm (0.020 in.). The 0.002 in. holes were about the limit of unaided eye detectability, and the 0.001 in. holes were virtually invisible. The larger size holes were readily discernible without visual magnification.

A microscopic photograph of the coined ends of inert detonator samples is shown in figure 2. The upper row, left to right, shows a good end, then hole diameters of 0.001 in. and 0.002 in. The second row, left to right, shows bottoms with hole diameters of 0.005 in., 0.010 in., and 0.015 in. Figures 3 4, 5, 6, 7, and 8 have the same sequence of flaws magnified approximately 50 times. Ten of the marked units are equal to 1 millimeter. These photos illustrate the difficulty in visually detecting flaws which are less than 0.005 in. but which should be readily detected by a microscopic optical system.

These inert detontors were immersed in water, according to the specification, to determine the quantitative relationship between the hole size and the amount of water absorbed. A second series of tests with inert detonators were carried out according to the method of helium-leak detection to determine leak rate characteristics; however, the quantitative determinations were limited to those inert detonators with 0.001 in. holes. This limitation was imposed first, because initial results with larger size holes were erratic, and second, the leak rates from detonators with large hole sizes would either indicate gross leakage or lead to misleading results, indicating properly sealed items when actually the helium could have been exhausted before the mass spectroscopic analysis (ref 2). Furthermore, the larger size holes would have been detected visually or instrumentally during process inspection and the detonators would have been rejected prior to being packed or tested. Thus, the probability of sampling a detonator having an undetected flaw, such as a large exterior hole or cut, would be minimal.

Additionally, punctured live detonators were subjected to the moistureproofness and functioning test to determine the change in sensitivity. These detonators were impacted with a pin fired from an air gun (ref 4) instead of being activated by the traditional ball drop method.

Molecular Flow Equation

Leak detection, according to the back pressuring technique, is conducted by external pressurization with tracer gas which enters a flawed container. Presence or absence of a leak and its size are detected by using a mass spectrometer to determine the mass flow of escaping gas. The equation governing leak rate can be formulated from a theory of gaseous molecular flow through an orifice. The following derivation is essentially obtained from the discussions of Howl and Mann (ref 3) and Turnbull (ref 5), wherein the three stages of pressurization, the elapsed time before testing, and the actual test itself contribute to the total mass flow rate.

Stage 1. Pressurization

During this stage, the item is immersed in the atmosphere of tracer gas. The mass flow rate into the item through a leak is given by the mean velocity of the gas and the pressure differential.

$$V(dp/dT) = C(A/M)^{1/2}(p_e - p) \tag{1}$$

p_e = external pressure of tracer gas

p = tracer gas in the item

A = absolute temperature

M = molecular weight of gas

Therefore

$$\int dp/(p_e - p) = (C/V)(A/M)^{1/2} \int dT$$

$$\ln (p_e - p) = -(C/V)(A/M)^{1/2}T + C_i$$

The integation constant $C_i = \ln p_e$ is obtained by setting $p = 0$ at $T = 0$. Thus the internal pressure p_1 at time T is given by:

$$\ln [(p_e - p_1)/p_e] = -(C/V)(A/M)^{1/2}T$$

$$(p_e - p)/p_e = \exp [-(C/V)(A/M)^{1/2}T]$$

$$p_1 = p_e \{1 - \exp [-(C/V)(A/M)^{1/2}T\} \tag{2}$$

Stage 2. Wait Time

After removal of external tracer gas pressure p_e, there is a time interval until the actual test with a mass spectrometer. During this time lapse, the mass flow rate of escaping gas out of the item through the leak is given by

$$-V (dp/dt) = C(A/M)^{1/2} p$$

Integration of this equation in proper form results in the expression for the tracer gas pressure p_2 in the item at wait time t. The constant of integration ($C_i = \ln p_1$) is obtained from the conditions: $t = 0$ at the initial wait time and $p = p_1$ (equation 2) for the internal pressure when $t = 0$.

$$\int dp/p = -(C/V)(A/M)^{1/2} \int dt$$

$$\ln p = -(C/V)(A/M)^{1/2} t + \ln p_1$$

$$p_2 = p_1 \exp\left[-(C/V)(A/M)^{1/2} t\right] \tag{3}$$

Stage 3. Actual Test

During the test, the leak rate is measured with a mass spectrometer. The mass flow of tracer gas is dependent on the internal gas pressure p_2 at wait time t (equation 3).

$$R = -V(dp/dt)$$

$$= -C(A/M)^{1/2} p_2 \tag{4}$$

$$= \text{measured leak rate}$$

define

L = leak size

= *one atmosphere of air leaking into a vacuum*

where

$$L = -V\ (dp/dt)$$

$$= -C\ (A/M_{air})^{1/2} p_o \tag{5}$$

p_o = one atmosphere

M_{air} = molecular weight of air = 30

Rewrite equation 5

$$L(M_{air})^{1/2}/p_o = -C(A)^{1/2} \tag{6}$$

Substitute equation 6 into the measured leak rate equation 4

$$R = L\,(M_{air}/M)^{1/2}\,(p_2/p_o) \tag{7}$$

Before making further substitutions, rewrite equations 2 and 3 using equation 6.

$$p_1 = p_e\,\{1-\exp\,[-(L/p_o)(M_{air}/M)^{1/2}\,(T/V)]\}$$

$$p_2 = p_1\,\exp\,[-(L/p_o)(M_{air}/M)^{1/2}\,(t/V)]$$

or

$$p_2 = p_e\,\{1-\exp\,[-(L/p_o)(M_{air}/M)^{1/2}\,(T/V)]\}\,\{\exp\,[-(L/p_o)(M_{air}/M)^{1/2}\,(t/V)]\} \tag{8}$$

Substituting equation 8 for p_2 into equation 7 results in

$$R = (L/p_o)(M_{air}/M)^{1/2}\,p_e\,\{1-\exp\,[-(L/p_o)(M_{air}/M)^{1/2}\,(T/V)]\}\,\{\exp\,[-(L/p_o)(M_{air}/M)^{1/2}\,(t/V)]\} \tag{9}$$

The measured leak rate R, therefore, is obtained from the parameters L and V inherent in each individual item, and from the variables p_e, T, and t which are controllable by the experimenter. Equation 9 is used in MIL-STD-883 for testing packaged electronic devices. In the "Fixed Method," a reject limit is given for leak rate R, which is measured with a mass spectrometer. For the "Flexible Method," a limit is placed on the equivalent standard leak rate L from which the limit on the measured leak rate R is calculated.

In MIL-STD-331A, Test 118 for sealed fuzes, reference is made to the article by Howl and Mann (ref 3); however, a leak rate limit of 1×10^{-8} atm cm^3 per sec is set as the reject level without resorting to equation 9.

If the leak size L is defined as the mass flow rate of helium at one atmosphere pressure escaping into a vacuum instead of being defined in terms of air, the ratio of molecular weights is one, and since $p_o = 1$, the measured rate R can be expressed by

$$R = Lp_e\,\{1-\exp\,[-(L/V)T]\}\,\exp\,[-(L/V)t] \tag{10}$$

This is the equation used by Briggs and Burnett (ref 6) to obtain their tables of leak rates for several leak sizes, volumes, bombing times, wait times, and at a five-atmosphere pressurization with helium. *(There are two discrepancies in*

their formula: the "bombing" time in the first bracket should be T, and the "bombing" pressure P is missing.)

Taking the logarithm of equation 10 results in

$$\ln R = \ln L + \ln p_e + \ln \{1-\exp [(-L/V)T]\} -(L/V)t \qquad (11)$$

When a given item is tested by the back pressurization method, at a given pressure p_e for a given time T, the first three terms on the right side of equation 11 remain constant. Therefore, the logarithm of the measured leak rate R is linearly related to the wait time t, and plotting the rates of the same item over several wait times should result in a straight line having a slope of -(L/V), or, in other words, the logarithm of the rate should decay linearly with time. Ideally, for all wait times with items of the same geometry, identical contents, and precisely drilled holes, the leak rates should lie on the same line.

A similar result occurs for equation 9 with logarithmic manipulations, except that the slope now represents

$$-(L/V)(M_{air}/M_{He})^{1/2}$$

where L is again defined by equation 4.

EXPERIMENTAL PROCEDURE

Weight Gain of Inert Detonators

Ten inert M55 detonators from a control group and 10 inerts each with precision-drilled holes of 0.005 cm (0.002 in.), 0.013 cm (0.005 in.), 0.025 cm (0.010 in.), 0.038 cm (0.015 in.), and 0.051 cm (0.020 in.) diameter were weighed on an analytical balance. They were then placed in separate beakers containing 7.62 cm (3 in.) of distilled water at the laboratory temperature which remained within 21.1 ± 5.6°C (70 ± 10°F) for 48 hours. Each simulated detonator was wiped dry of exterior moisture, then reweighed. The difference in weight before and after water immersion was assumed to be due to moisture pickup.

Live Detonator Moistureproofness Test

Live detonators were carefully punctured with a needle, and the hole dimensions were determined by means of a measuring microscope. The detonators with 0.002 in. and 0.005 in. diameter holes were immersed in 7.62 cm (3 in.) of distilled water maintained at 21.1 ± 5.6°C (70 ± 10°F) for 48 hours. The 50% fire

level was determined for a control group with an "up-and-down" type test using air pressure-actuated firing pins. The 50% fire levels were then determined by the same method for those detonators subjected to water immersion.

Helium-Leak Test

Helium-leak tests by the bombing technique, carried out on inert detonators at the Varian Associates, Lexington Vacuum Division, Lexington, Massachusetts are described in the appendix. Samples of simulated M55 detonators were placed in evacuable metal containers and evacuated with a mechanical pump. The pump was then cut off from the system and the containers were pressurized with four atmospheres of helium. Separate samples were maintained at this pressure for 1, 2, or 3 hours. The individual samples were then placed in the evacuation port of a helium-leak detector. After rapid evacuation of the air from this port, the sample was switched to a mass spectrometer which measured the rates of helium leak.

RESULTS AND DISCUSSION

Moisture Absorption

Results of the water immersion tests of inert detonators with the specified hole dimensions, as well as of inert detonators without flaws serving as reference standards, are shown in tables 1 through 6. The controls or reference standards (table 1) appear to have lost weight ranging from 0.1 mg to 0.4 mg which is probably due to experimental errors. The logical errors should have been a tendency to gain weight because of moisture on the outer surface rather than a loss of weight which implies a loss in material. This is unlikely since they appeared well sealed when visually inspected. Furthermore, an extra coat of clear lacquer was p inted on the old lacquer to insure a seal. The detonators with 0.002 in. holes (table 2) showed weight "gain" ranging from -0.3 mg to 1.4 mg. The reasons for loss of weight in the controls and the inerts with 0.002-in. holes remain unanswered. Only the inerts with the largest size hole (0.020 in.) (table 6) showed a reasonably uniform gain in weight where the average was 3.9 mg with a standard deviation of 0.4 mg.

The mean and range of weight gains with respect to hole sizes are plotted in figure 9. Although the range is large, the trend is obvious that more moisture is absorbed as the holes become larger, which is to be expected. It was hoped that the experimental results would provide quantitative information rather than a simple confirmation of a logical assumption. Larger sample sizes are not expected to provide greater precision in the values since other factors, such as capillary action, diffusion phenomena, and intersurface forces, exert considerable effect on the ability of water to penetrate into the interior of the item with the hole dimensions being studied.

The linear regression line for the averages of experimental values obtained for the various hole sizes is given by $y = -0.19 + 7.98\ x$, where y is the weight gain in milligrams after 48 hours' immersion in 5.08 cm to 7.62 cm (2 to 3 in.) of water maintained at 21.1° ± 5.5°C (70° ± 10°F), and x is the hole size in mm. Accordingly, the item would absorb 0.01 mg of water for a 0.0254 mm (0.001 in.) hole. The theoretical line predicts a negative value of -0.19 mg for an item without a hole; however, since an item without a flaw should neither gain nor lose weight, the negative y intercept for the linear regression line shows the inherent errors of the experimental method. The line of best fit should be of the form $y = 0 + bx$. In addition, the wide range in data points for a given diameter indicates the difficulty of these experiments where the weight gain is in the mg range.

For an M55 detonator, loaded according to drawing 8798331 with 85 mg of explosives (15 mg NOL-130, 51 mg lead azide, and 19 mg RDX), the permissible level of moisture is 0.36 mg according to specifications applicable to these chemical constituents. The empirical equation cited above indicates that 0.4 mg of moisture would be absorbed by a detonator with a 0.002 in. hole when tested according to the method required in the detonator specification. Thus, moisture in excess of the acceptable level can penetrate into an explosive composition when its container is perforated with punctures or holes 0.002 in. diameter or larger.

Live Detonator Tests

Live detonators with 0.002 in. holes on the coined end of the aluminum cup were subjected to the moistureproofness test according to MIL-D-14978A. The functioning test of the detonators was carried out with a pressure-actuated firing pin instead of the ball-drop test. The results are shown in table 7. The data indicate that 50% firing velocity for the control group (the detonators which were not subjected to moisture) was 390 cm (12.8 ft) per sec. The 50% firing velocities for detonators with 0.002 in. and 0.005 in. holes were 479 cm (15.7 ft) per sec and 451 cm (14.8 ft) per sec. Although the test indicated that more energy was required to fire detonators with the smaller (0.002 in.) hole size than the larger (0.005 in.) one, the tendency was a reduction in sensitivity of the defective detonators. In addition, if the detonators did not fire on the first trial, a second needle was fired at 640 cm (21 ft) per sec to destroy the detonators. While the controls all fired at this energy input, some of the defectives only partially fired on the second trial.

Helium Leak Rates

The helium-leak test using the bombing technique on control samples which appeared flawless and had well lacquered output ends, registered various leak rates ranging from zero to high values, which indicated either experimental difficulties or true leakers. Although some tests were carried out on samples with holes greater than 0.001 in., only the characteristics of samples with the smallest holes were followed in detail because of greater consistency in experimental data.

Results of tests on samples with 0.001 in. holes are shown in tables 8, 9, and 10, and in figure 10. Leak tests were carried out after bombing (pressurization) times of 1, 2, and 3 hours. Leak rates for several elapsed times (wait times) were measured for individual items pressurized for 1 hour; leak rates for individual items pressurized for 3 hours were taken at 2 elapsed times; and those items pressurized for 2 hours were tested for leaks only once. The data were recorded for wait times of up to 1,860 seconds (31 minutes). The data points for 1-hour pressurization time were connected since they represent several readings per item; however, the data points for 2- and 3-hour pressurization times were not connected since they represent one and two readings per item, respectively. The three straight lines (open circles) in figure 10 were obtained from the data of Briggs and Burnett (ref 6). They represent leak rates R calculated from equation 11 for items with a volume of 10^{-3} cm^3, which were "bombed" for time T of 1 hour at a pressure of 5 atmospheres, and which had actual leak rates L of 10^{-6}, 10^{-7}, and 10^{-8} std cm^3 per sec.

Although, according to equation 11, a linear relationship should result from a plot of the logarithm of leak rates against wait time, the experimental data do not decrease linearly but asymptotically toward the sensitivity limit of the mass spectrometer, as can be seen from figure 10. This phenomenon probably holds true for any item filled with material having a large surface area as contrasted to an item with a "clean" volume such as packaged electronic components. The theoretical relationship, according to the formula of Briggs and Burnett for small volumes (0.01 cm^3) and actual leak rates L of 10^{-5} std cm^3 per sec, should be linear beyond the time for which data was collected in the present study. Further, the bombing time should not vary the indicated leak rate.

The volume of the detonator container, calculated from the dimensions of figure 1, is approximately 0.03 cm^3; however, the free volume is effectively eliminated by the three highly compressed explosive components. Thus, the helium can only be occluded in a much smaller interstitial volume. The conductance, or the actual leak rate L, was measured by opening, emptying, and sealing the punctured end into the wall of the vacuum system. The pressure P in the system due to the air flow through the 0.001 in. hole was measured and multiplied by the pumping speed of the vacuum pump. This was the conductance through an unobstructed hole giving the upper limit to the flow. The same measurements made with the detonators open and the material intact gave the lower limit to the flow. The upper limit was about 0.4 std cm^3 per sec and the lower limit, 1.3×10^{-2} std cm^3 per sec. It is obvious from the present data that the actual leak rate L was much faster (0.40 to 0.013 std cm^3 per sec) than that for the working tables of Briggs and Burnett (10^{-8} to 10^{-5} std cm^3 per sec).

On the other hand, if the logarithm of the leak rates is plotted against the logarithm of time (fig. 11) instead of time itself, the result is a series of straight lines for the data obtained for individual items pressurized for 1 hour (table 8) and whose leak rates were determined for a number of wait times extending over a period of more than 300 seconds. The data for 1-hour pressurization have been plotted since three data points per item were available (except for data points (30, 10) and (245, 1.6), while only one data point and two data points per item were obtained for 2 and 3 hours' pressurization, respectively.

Assuming that the foregoing log-log linearity describes the kinetics of the leakage, the logarithm of all the leak rate data of tables 8, 9, and 10 were plotted against the logarithm of wait times (fig. 12). The filled circles are values for the items pressurized for 1 hour, the x's for 2 hours, and triangles for 3 hours. The line of best fit is represented by:

$$\log R = -4.58 - 1.31 \log t$$

Although there is some scatter, the values fall neatly in a narrow band around the linear regression line. The fact that these data were obtained for three pressurization times, and yet overlap, indicates that the pressurization time T of equations 9, 10, and 11 does not affect the leak rate. In other words, the items are saturated within 1 hour or less and long pressurization times are not necessary. The linearity of the log-log relationship is analogous to the low temperature (30° to 495°C) results of helium desorption from mica (ref 13).

The implication of leak-rate results is that equation 11, derived from a simple consideration of molecular flow, does not adequately describe helium leakage from a small volume containing material with considerable surface area, and that the desorption phenomenon deserves closer scrutiny as a possible explanation for the present results.

Subsequent to completion of the inert detonator tests, under a separate program, live detonators with diverse sealing configurations were helium leak-tested and output tested after water immersion (ref 8). The leak test results are shown in tables 11, 12, and 13. The items tested were 50 detonators each from:

1. A normal production lot of Lone Star Army Ammunition Plant M55 detonators

2. Loaded detonators without lacquer coating on the closing discs

3. Loaded detonators with uncoated closing discs ultrasonically sealed to the crimp

4. Detonator with chromated green discs ultrasonically sealed to the crimp

The leak test results from the normal production lot are not tabulated herein since they all showed zero leakage from wait times of 65 to 2,555 seconds. Although zero leakage could have been due to gross leakers, this was unlikely since 50 individual detonators were tested. The assumption of zero leakage is further justified from the fact that the other three series of detonators demonstrated detectable leak rates over approximately the same time span.

These live detonator tests were carried out somewhat differently from the inert detonator tests. Fifty detonators from each series were individually tested at different wait times after "bombing" rather than testing one detonator's over the entire time period. Therefore, the leak configuration was not

constant. However, the procedure was consistent with the real-life situation wherein individual items would be tested. In addition, the helium leak from the live detonators was reported in relative rates. It is assumed that the absolute rates ranged from 10^{-7} to 10^{-9} std cm^3 per sec, as in the case for the inert detonators; otherwise, the helium from the faulty detonators should have been exhausted much sooner.

A plot of log R vs time, not unexpectedly, also resulted in a parabolic curve. The log R vs log t curve is shown in figure 13. Several interesting features emerge. The detonators with ultrasonically sealed, chromated discs (squares) and the unlacquered, unwelded detonators (triangles) leaked at nearly the same rates in the interval 100 to 700 seconds. The detonators with ultrasonically welded, plain discs (circles) leaked at lower rates than the other two types over the same time span. Beyond approximately 500 seconds, the scatter of the rates for the three series of detonators overlap and are not distinguishable. Since these were all leakers, there is a wide band of approximately five orders of magnitude for leak rates at any one point of wait time.

Although testing the inert and live detonators varied, comparison is inevitable. When figure 13 (live detonators) is overlaid on figure 12 (inerts), the chromated sealed disc (squares) and the unsealed, unlacquered detonator (triangles) lie near the regression line obtained previously with the data from inert detonators. The ultrasonically sealed, plain disc detonators, however, leak at much reduced rates and deviate widely from the line. This would indicate that a wide range of leak rates can be expected from leaky detonators.

The functioning and output tests after water immersion (table 14) indicated that 96% of the normal detonators were satisfactory while only 11% of the unwelded, unlacquered detonators were satisfactory. Only 9% of the bare disc, ultrasonically welded detonators and 36% of the chromated, ultrasonically welded detonators satisfactorily passed the test.

A consideration of leak test methods now in use and of results of this study offers two options for establishing a leak rate method as a substitute for the present moistureproofness test:

First, an absolute leakproof limit can be placed on acceptable detonators. In this case, no leak, within the sensitivity of the mass spectrometer, would be permissible. However, a second "gross leak" test may be required since the absence of a leak may be due to large exterior defects through which helium is completely exhausted before the leak rate is measured.

Second, a leak rate limit may be placed on an unacceptable leak as is the case for electronic packaging and for fuzes. However, in this case, the range or band-width of leak rates found for a variety of detonator exterior defects (unsealed, poorly sealed, and 0.001 in. holes) must be more accurately defined. In addition, the relationship between leak rates within this band and moistureproofness failure rates must be firmly established.

CONCLUSIONS

Results of this program indicated that M55 detonators with 0.005 cm (0.002 in.) diameter holes, which are barely visible with the unaided eye, can be affected by moisture when subjected to the test specified in MIL-D-14978. The weight gains due to moisture absorption by detonators with 0.002 in. and 0.001 in. diameter holes was not consistent from one sample to the next and, therefore, a relationship between puncture diameters and moisture pickup cannot be accurately predicted for these minute but critical flaws. Impact sensitivity of detonators can be adversely affected when detonators are immersed in water if the metallic exterior has a puncture of 0.002 in. diameter.

Although the helium leak detector has a reasonably viable means of detecting punctures equivalent to 0.001 in. diameter by means of the back-pressuring technique, and since the results do not demonstrate the linearity expected from equation 11 which is used in MIL-STD-331 for leak testing fuzes, the accept/reject criterion of the standard is not necessarily applicable to the M55 detonator. Furthermore, because of the small volume of the detonator cup and further reduction of the free volume by compressive loading with explosive components, only a small amount of helium can be introduced. Since helium depletes rapidly, only a small amount will be observed in a leak test. Thus, such a test, when developed for a requirement which is applicable, must be carried out within a reasonable wait time before the gas is completely exhausted.

RECOMMENDATIONS

Since the inspection--whether visually or electronically--of loaded detonators can detect flaws such as cuts, tears, and holes of 0.005 cm (0.002 in.) width or diameter, and since both the test for weight gain due to moisture and the firing test indicated that moisture can affect detonators with this size aperture, the problem of moistureproofness or leakage in a detonator container is in apertures of 0.002 in. or less. An electro-optical device is planned for inspection of all detonators from the loading machine, and it is anticipated that defects on the metallic exterior will escape detection less frequently than flaws on the lacquer coated crimp end. The lacquer painting is a separate operation and the inspection will be visual. Flaws in the lacquer, such as pin-holes and defective interfacial adhesion of lacquer to the metal surface, may not be obvious to the unaided eye. Thus, moisture penetration would likely occur, if at all, from the lacquered end during the water immersion test.

Moisture penetration from the liquid phase (immersion of item under water) through an aperture of the size in the present study undoubtedly occurs by means of capillary action and depends on surface phenomena rather than on a fluid flow mechanism. Furthermore, since the M55 detonator is not normally stored under water, but in the atmosphere with exposure to water in the vapor phase, continued investigation should also include constant temperature and humidity tests.

The helium-leak test on 0.001 in. diameter holes and the functioning test after water immersion on 0.002 and 0.005 in. diameter holes, demonstrated that

the leak test can detect defects which are virtually invisible and that moisture penetration through 0.002 in. diameter holes can affect the detonator sensitivity. However, an upper limit on an acceptable leak, not as yet established, is necessary prior to inclusion in any specification. The validity of the results should be confirmed with further detailed experiments and larger sample sizes. Experiments with live detonators are desirable; however, the hazards associated with these items pose a problem. The necessity of retesting for gross leaks when no leaks are indicated by fine leak testing (helium-leak test) poses a redundancy problem that requires resolution. In conjunction with the helium-leak test, long term storage tests of live detonators, as well as long-term, continuous testing to determine reliability of the method, are required.

The further testing and resolution of the foregoing problems must be accomplished before consideration is given to implementing the helium leak test to assure the exterior integrity of the M55 detonator.

REFERENCES

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Table 1. Moisture test on inert detonators (control, no holes)

Specimen no.	Water immersion: Wt before (g)	Water immersion: Wt after (g)	Change in wt (g)
1	0.1155	0.1155	0.0000
2	0.1148	0.1147	-0.0001
3	0.1167	0.1166	-0.0001
4	0.1139	0.1137	-0.0002
5	0.1124	0.1122	-0.0002
6	0.1128	0.1125	-0.0003
7	0.1147	0.1146	-0.0001
8	0.1170	0.1167	-0.0003
9	0.1074	0.1070	-0.0004
10	0.1151	0.1147	-0.0004
			$\bar{X}$ = -0.0002
			s = 0.0001

Table 2. Moisture test on inert detonators with 0.005 cm (0.002 in.) holes

Specimen no.	Water immersion: Wt before (g)	Water immersion: Wt after (g)	Change in wt (g)
1	0.1060	0.1062	0.0002
2	0.1060	0.1069	0.0009
3	0.1066	0.1069	0.0003
4	0.1063	0.1077	0.0014
5	0.1076	0.1073	-0.0003
6	0.1085	0.1086	0.0001
7	0.1062	0.1062	0.0000
8	0.1073	0.1070	-0.0003
9	0.1070	0.1070	0.0000
10	0.1148	0.1148	0.0000
			$\bar{X}$ = 0.0002
			s = 0.0005

Table 3. Moisture test on inert detonators with 0.013 cm (0.005 in.) holes

Specimen no.	Water immersion Wt before (g)	Wt after (g)	Change in wt (g)
1	0.1028	0.1040	0.0012
2	0.1165	0.1175	0.0010
3	0.1099	0.1107	0.0008
4	0.1050	0.1057	0.0007
5	0.1060	0.1063	0.0003
6	0.1071	0.1073	0.0002
7	0.1098	0.1103	0.0005
8	0.1042	0.1054	0.0012
9	0.1071	0.1082	0.0011
10	0.1146	0.1174	0.0028
			$\bar{X}$ = 0.0009
			s = 0.0007

Table 4. Moisture test on inert detonators with 0.025 cm (0.010 in.) holes

Specimen no.	Water immersion Wt before (g)	Wt after (g)	Change in wt (g)
1	0.1039	0.1056	0.0017
2	0.1157	0.1170	0.0013
3	0.1108	0.1120	0.0012
4	0.1065	0.1086	0.0021
5	0.1040	0.1056	0.0016
6	0.1060	0.1089	0.0029
7	0.1070	0.1079	0.0009
8	0.1067	0.1088	0.0021
9	0.1040	0.1063	0.0023
10	0.1055	0.1060	0.0005
			$\bar{X}$ = 0.0017
			s = 0.0007

Table 5. Moisture test on inert detonators with 0.038 cm (0.015 in.) holes

Specimen no.	Water immersion Wt before (g)	Wt after (g)	Change in wt (g)
1	0.1049	0.1085	0.0036
2	0.1053	0.1068	0.0015
3	0.1057	0.1068	0.0011
4	0.1044	0.1060	0.0016
5	0.1047	0.1084	0.0037
6	0.1052	0.1068	0.0016
7	0.1051	0.1098	0.0047
8	0.1039	0.1095	0.0056
9	0.1031	0.1082	0.0051
10	0.1049	0.1062	0.0013
			$\bar{X}$ = 0.0029
			s = 0.0018

Table 6. Moisture test on inert detonators with 0.051 (0.020 in.) holes

Specimen no.	Water immersion Wt before (g)	Wt after (g)	Change in wt (g)
1	0.1149	0.1185	0.0036
2	0.1135	0.1172	0.0037
3	0.1136	0.1180	0.0044
4	0.1116	0.1155	0.0039
5	0.1109	0.1151	0.0042
6	0.1099	0.1130	0.0031
7	0.1142	0.1182	0.0040
8	0.1139	0.1173	0.0034
9	0.1117	0.1158	0.0041
10	0.1135	0.1180	0.0045
			$\bar{X}$ = 0.0039
			s = 0.0004

Table 7. Water immersion test results with live M55 detonators

Sample[a]	50% fire level[b] (ft/sec)	Range (ft/sec)	Failure rate[b] at 21 ft/sec
A	12.8	11.7 - 13.9	0
B	15.7	7.1 - 21.3	2/34
C	14.8	11.6 - 17.4	3/33

[a] Sample types:

A = Normal detonators.
B = Detonators with 0.005 cm (0.002 in.) holes.
C = Detonators with 0.013 cm (0.005 in.) holes.

[b] Fired with air pressure-actuated 0.194-g needles.

Table 8. Helium leak rates (std $cm^3 \times 10^8$ per sec) for 1-hour pressurization

Wait time (sec)	Sample no. 1	2	3	4	5	6
30	10	-	-	-	-	-
70	-	6	-	-	-	-
105	-	-	12	-	-	-
140	-	-	-	3	-	-
170	-	-	-	-	4	-
210	-	-	-	-	-	1.6
245	1.6	-	-	-	-	-
280	-	1.5	-	-	-	-
330	-	-	2.4	-	-	-
350	-	-	-	1.0	-	-
375	-	-	-	-	1.2	-
405	-	-	-	-	-	0.7
465	-	0.8	-	-	-	-
480	-	-	1.4	-	-	-
510	-	-	-	0.6	-	-
530	-	-	-	-	0.7	-
555	-	-	-	-	-	0.5

Table 9. Helium leak rates (std $cm^3 \times 10^8$ per sec) for 2-hour pressurization

Wait time (sec)	Sample no. 1	2	3	4	5	6
300	0.9	-	-	-	-	-
720	-	0.5	-	-	-	-
1000	-	-	0.1	-	-	-
1320	-	-	-	0.2	-	-
1620	-	-	-	-	0.2	-
1860	-	-	-	-	-	0.1

Table 10. Helium leak rates (std $cm^3 \times 10^8$ per sec) for 3-hour pressurization

Wait time (sec)	Sample no. 1	2	3	4	5
120	6.0	-	-	-	-
150	-	5.4	-	-	-
210	-	-	7.2	-	-
240	-	-	-	2.6	-
260	-	-	-	-	2.5
280	2.0	-	-	-	-
310	-	2.1	-	-	-
340	-	-	2.2	-	-

Table 11. Loaded M55 detonators without lacquer coating on disc

Detonator no.	Relative leak rate*	Wait time (sec)	Detonator no.	Relative leak rate*	Wait time (sec)
1	90	60	26	3	1,335
2	35	105	27	2	1,385
3	27	150	28	1	1,430
4	15	190	29	2	1,470
5	15	235	30	1	1,510
6	13	265	31	1	1,550
7	9	305	32	1	1,595
8	8	355	33	1	1,640
9	8	395	34	1	1,690
10	8	435	35	1	1,730
11	7	495	36	1	1,760
12	10	640	37	2	1,835
13	7	690	38	1	1,880
14	6	745	39	1	1,925
15	5	800	40	2	1,960
16	6	855	41	0	1,995
17	4	900	42	0	2,035
18	5	945	43	0	2,080
19	4	990	44	0	2,115
20	3	1,035	45	1	2,115
21	4	1,080	46	0	2,200
22	4	1,135	47	0	2,235
23	3	1,225	48	0	2,275
24	2	1,265	49	1	2,310
25	2	1,305	50	0	2,350

* Arbitrary units.

Table 12. M55 detonator with ultrasonically welded uncoated closing disc

Detonator no.	Relative leak rate*	Wait time (sec)	Detonator no.	Relative leak rate*	Wait time (sec)
1	25	65	26	1	1,639
2	16	115	27	1	1,684
3	8	165	28	1	1,738
4	6	205	29	1	1,789
5	5	226	31	1	1,831
6	5	278	31	1	1,881
7	5	336	32	1	1,923
8	4	421	33	1	1,950
9	4	481	34	0	2,008
10	4	535	35	0	2,065
11	4	591	36	0	2,115
12	4	652	37	0	2,157
13	3	731	38	0	2,211
14	3	788	39	0	2,266
15	4	845	40	0	2,318
16	-	964	41	0	2,368
17	3	1,014	42	0	2,425
18	3	1,074	43	0	2,480
19	-	1,134	45	0	2,560
20	1	1,239	45	-	2,560
21	2	1,344	46	3	2,611
22	2	1,399	47	0	2,670
23	2	1,454	48	3	2,725
24	2	1,507	49	0	2,779
25	1	1,589	40	0	2,820

* Arbitrary units.

Table 13. M55 detonator with ultrasonically welded green chromated disc

Detonator no.	Relative leak rate*	Wait time (sec)	Detonator no.	Relative leak rate*	Wait time (sec)
1	19	172	26	1	1,280
2	15	240	27	1	1,300
3	20	285	28	1	1,380
4	12	320	29	0	1,425
5	12	365	30	0	1,470
6	10	405	31	1	1,520
7	8	440	32	1	1,560
8	7	480	33	0	1,605
9	7	520	34	0	1,645
10	3	560	35	0	1,690
11	5	590	36	0	1,740
12	3	645	37	0	1,775
13	2	690	38	1	1,820
14	5	735	39	1	1,875
15	2	785	40	0	1,915
16	4	835	41	0	1,960
17	3	875	42	0	2,000
18	3	920	43	0	2,050
19	3	965	45	0	2,100
20	2	1,015	45	0	2,155
21	2	1,060	46	0	2,210
22	2	1,110	47	0	2,260
23	2	1,160	48	0	2,310
24	1	1,205	49	0	2,370
25	2	1,250	50	0	2,415

* Arbitrary units

Table 14. Results from moistureproofness and output tests on M55 detonators

Central Testing Building Report
Lone Star Army Ammunition Plant
11 November 1980

Report no. 3995
Lot no. LS-80K326-018
Current production

No. of detonators	Disc indentation (in.)
4	0.000
1	0.015
3	0.016
10	0.017
27	0.018
35	0.019
13	0.020
3	0.021
2	0.022
2	0.023

Report no. 3996
Lot no. KNE-1
Clear discs (unlacquered, unwelded)

No. of detonators	Disc indentation (in.)
89	0.000
2	0.015
3	0.016
5	0.018
1	0.020

Report no. 3997
Lot no. LS79E0015418
Green chromated discs (welded)

No. of detonators	Disc indentation (in.)
64	0.000
3	0.016
6	0.017
11	0.018
9	0.019
7	0.020

Report no. 3998
Lot no. KNE-1
Clear discs (welded)

No. of detonators	Disc indentation (in.)
91	0.000
1	0.014
1	0.015
2	0.016
3	0.018
1	0.019
1	0.020

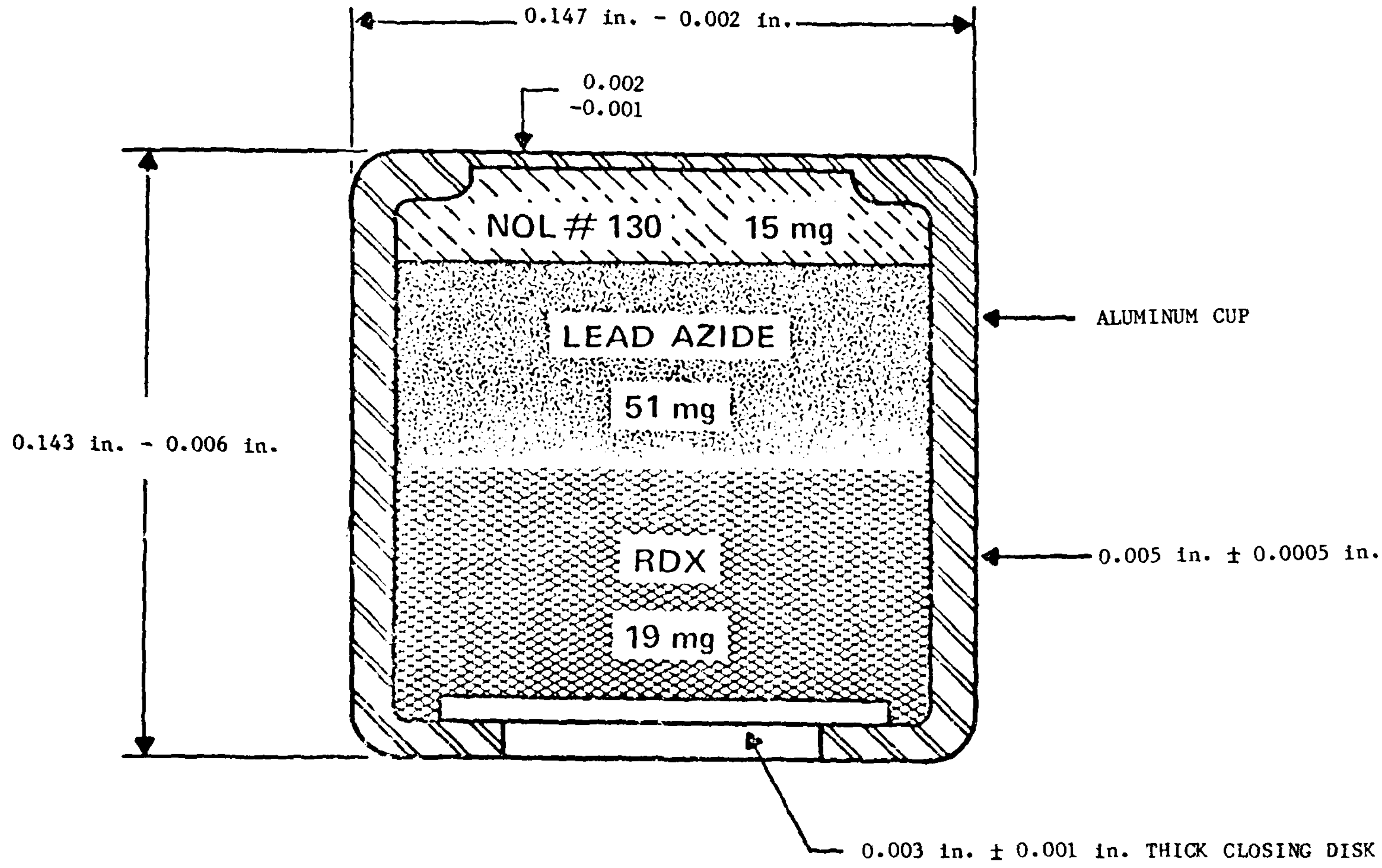

Figure 1. M55 detonator

Figure 2. Coined end of inert M55 detonators

Figure 3. Inert M55 detonator without puncture

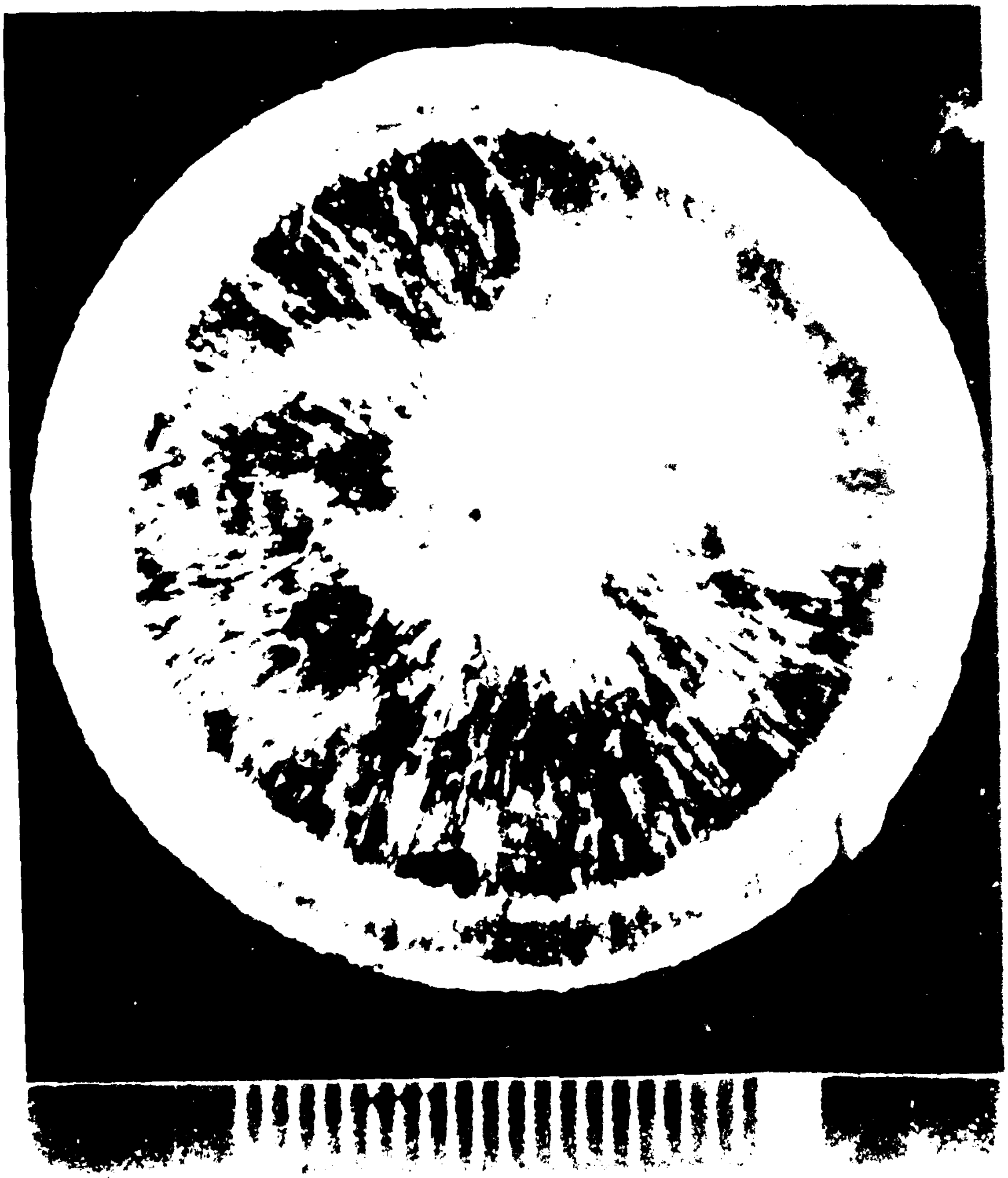

Figure 4. Inert M55 detonator with 0.0025 cm (0.001 in.) hole

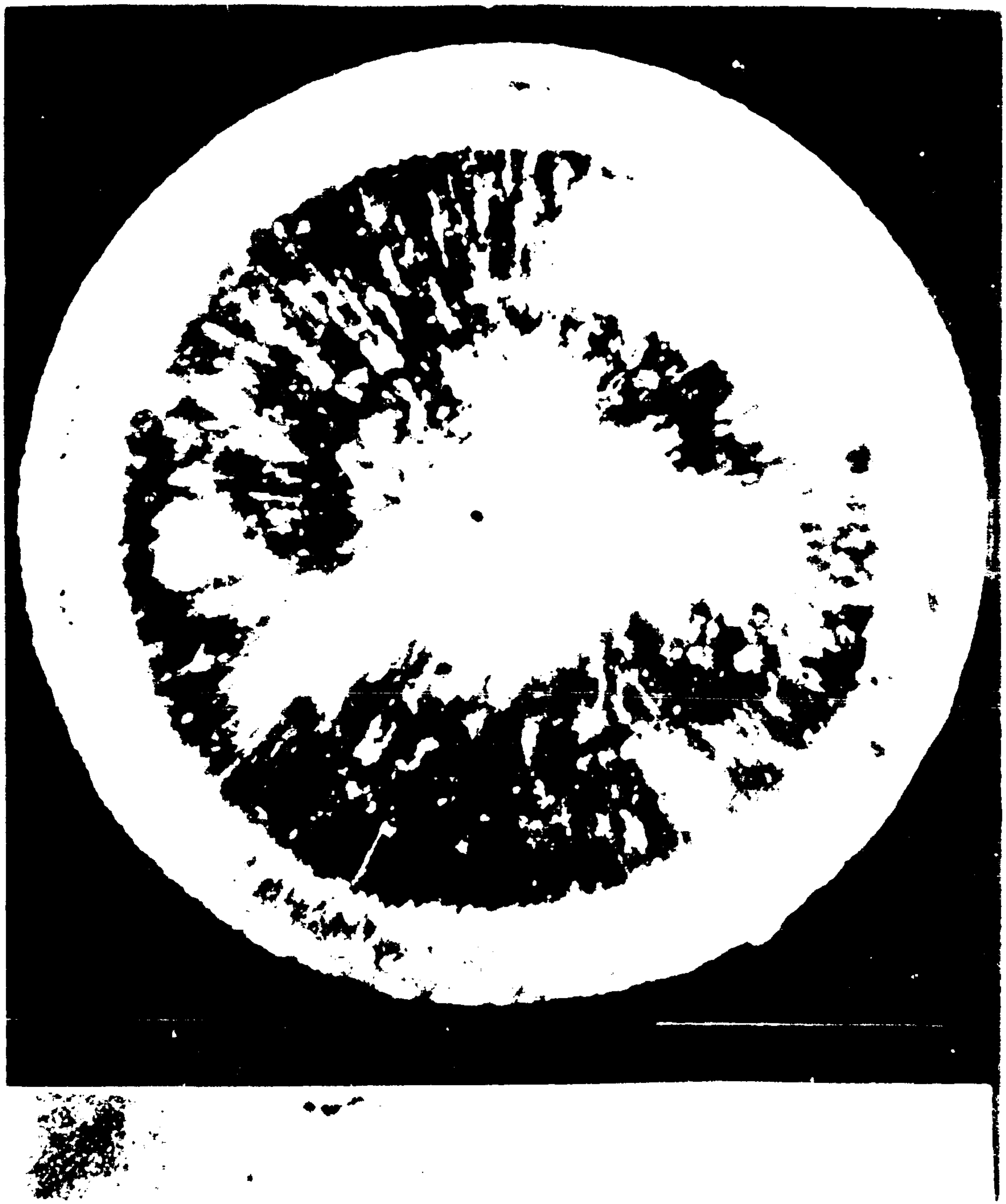

Figure 5. Inert M55 detonator with 0.005 cm (0.002 [illegible]

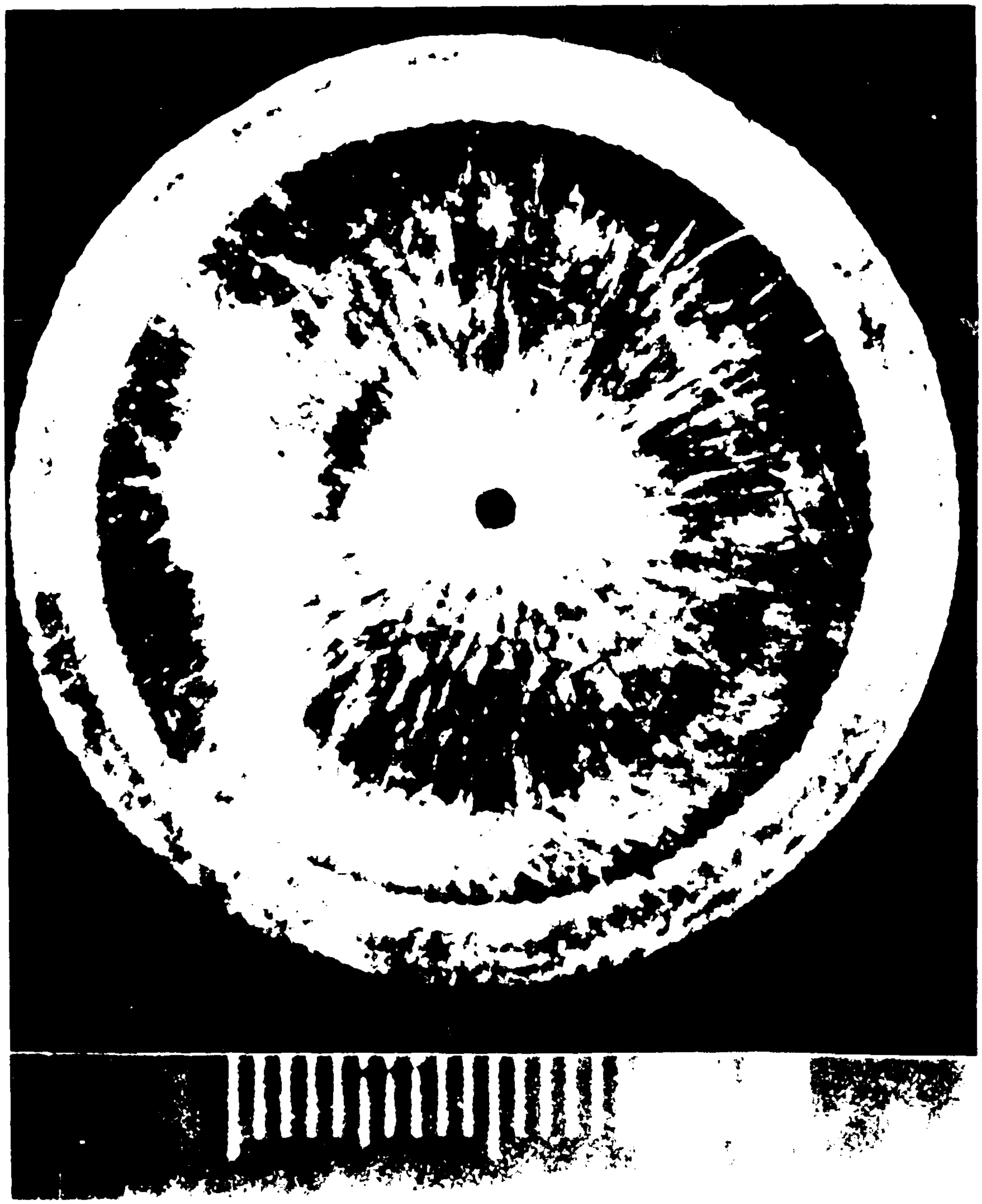

Figure 6. Inert M55 detonator with 0.013 cm (0.005 in.) hole

Figure 7. Inert M55 detonator with 0.02[illegible] cm [illegible] hole

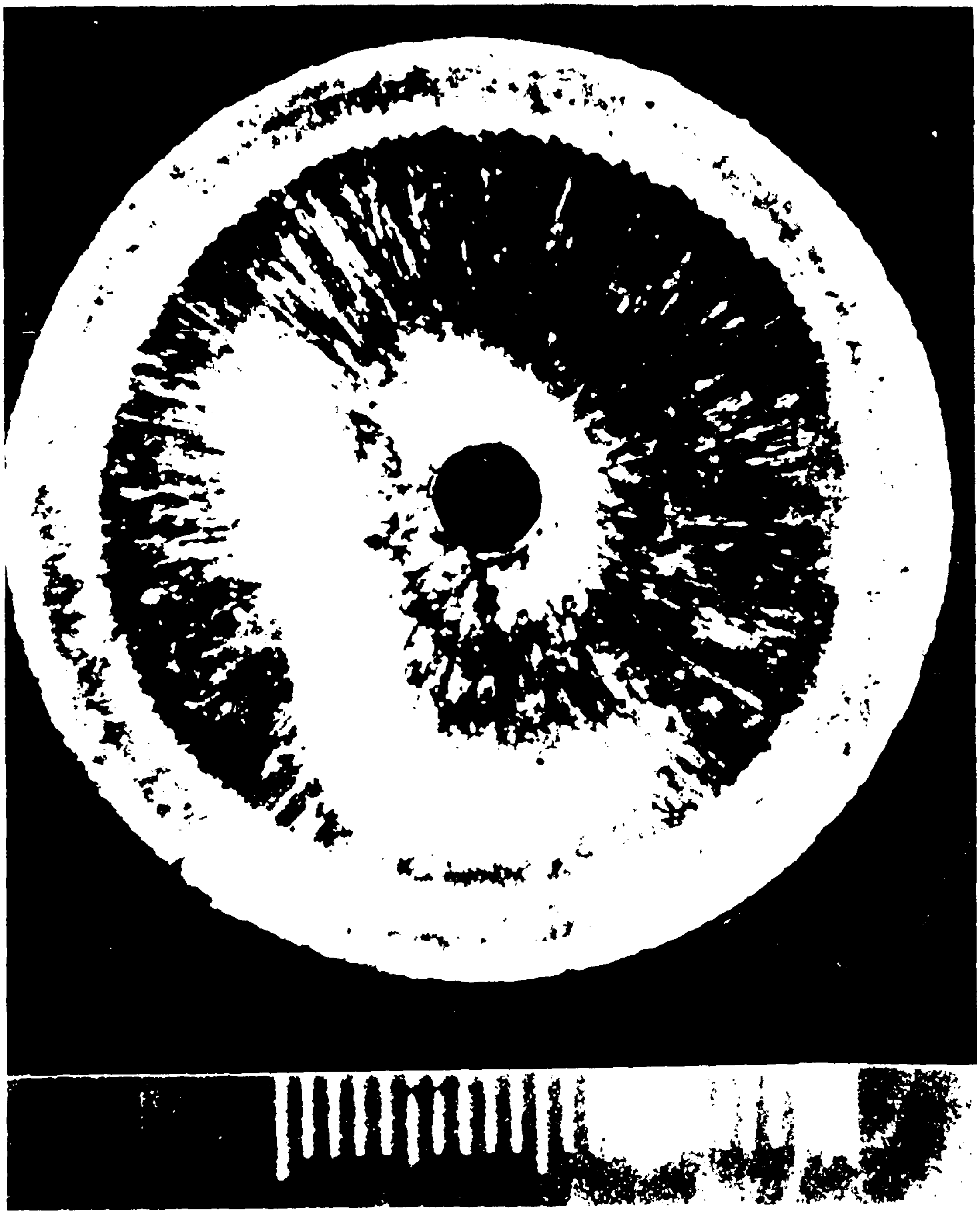

Figure 8. Inert M55 detonator with 0.038 cm (0.015 in.) hole

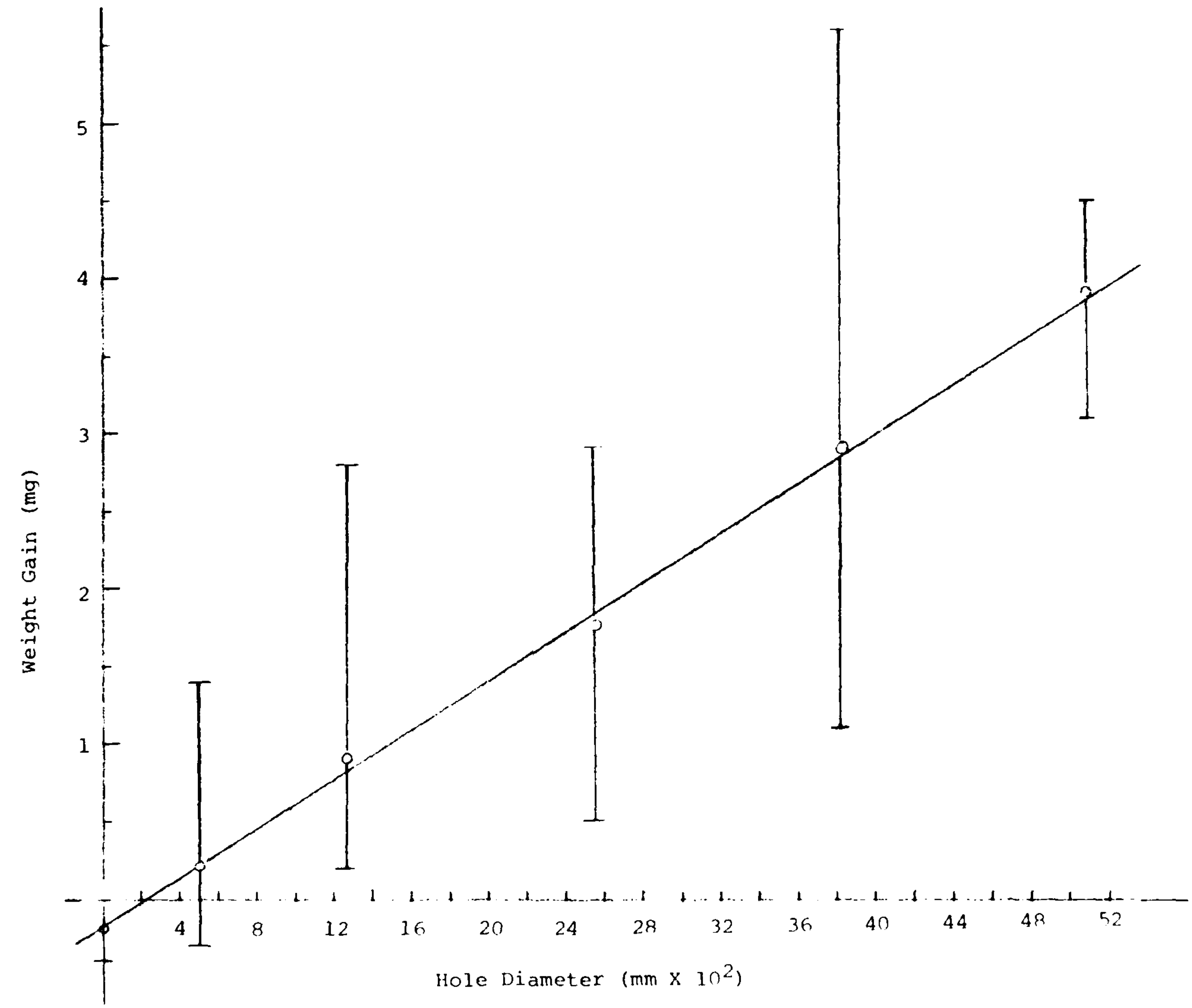

Figure 9. Average weight gain after water immersion

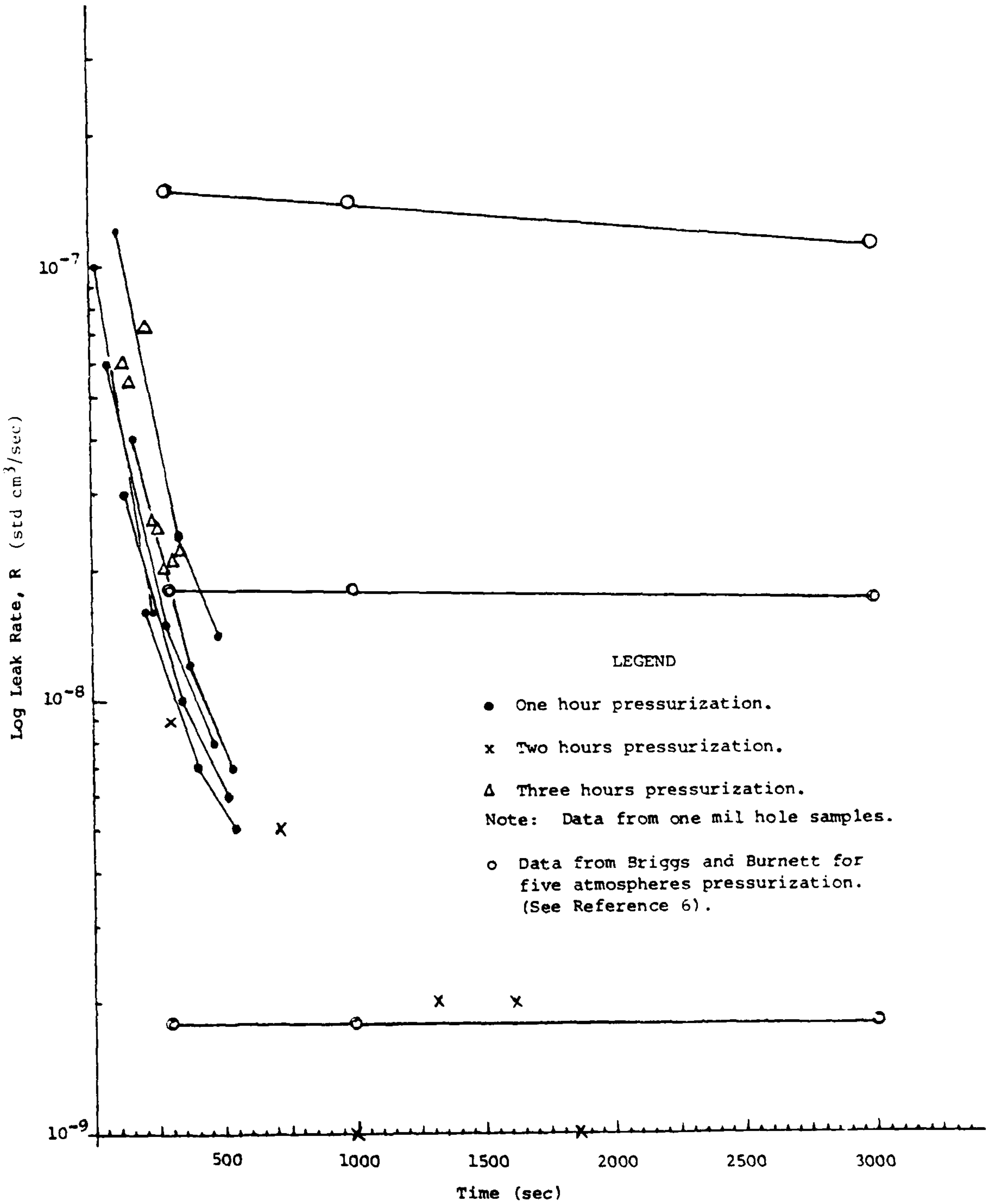

Figure 10. Time dependence of leak rate

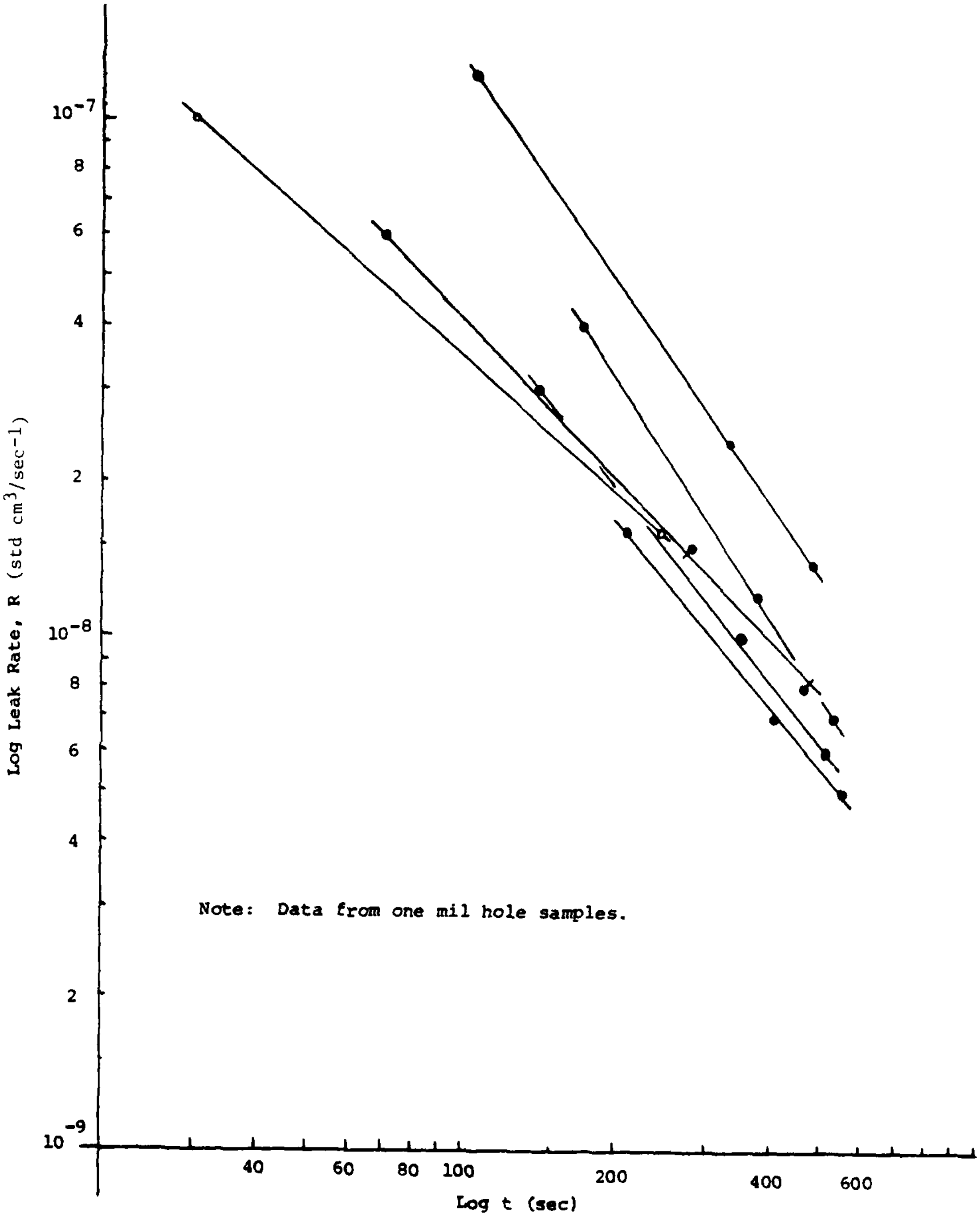

Figure 11. Logarithmic relationship of leak rate and wait time for one hour pressurization

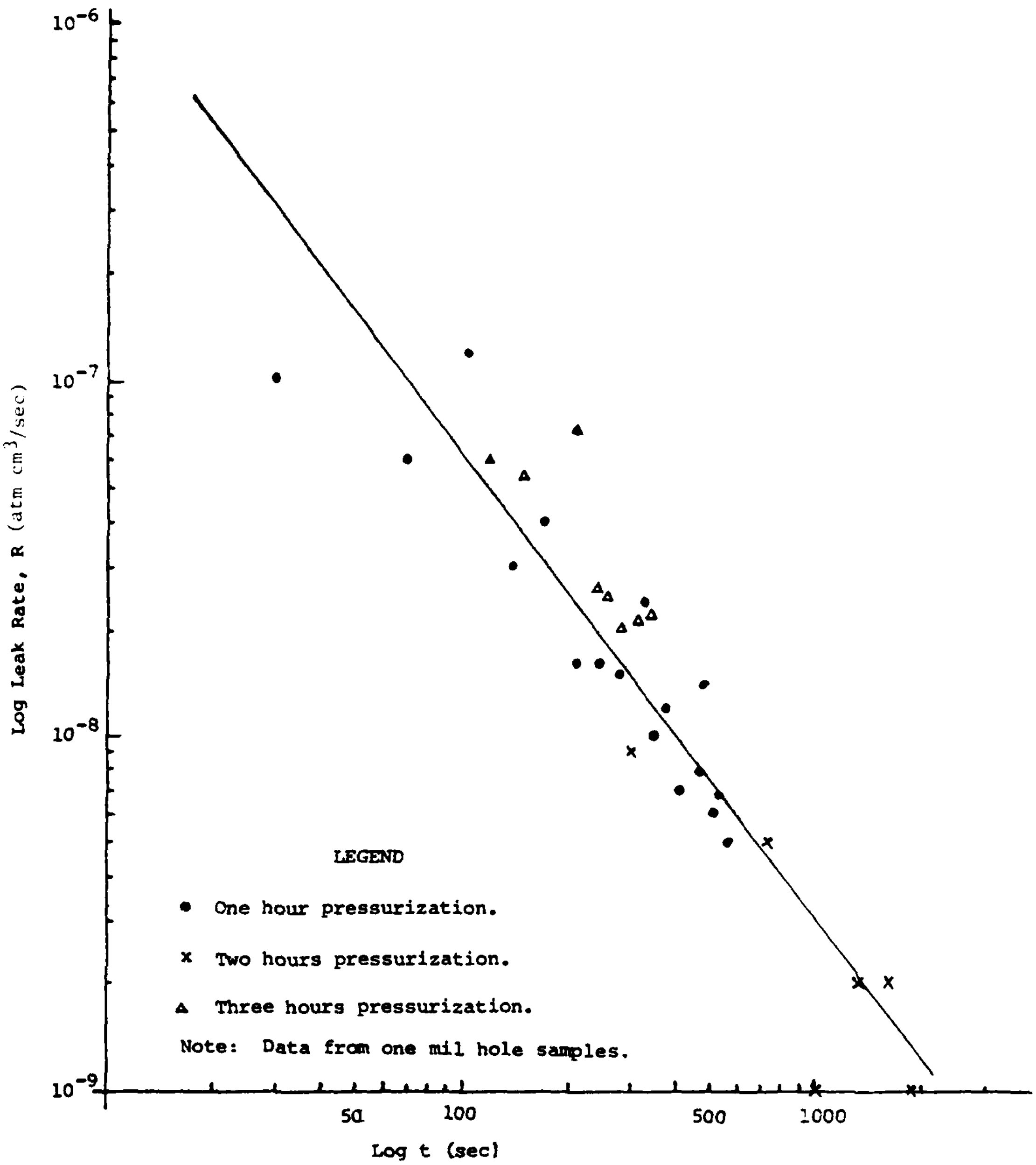

Figure 12. Logarithmic relationship of leak rates and wait times

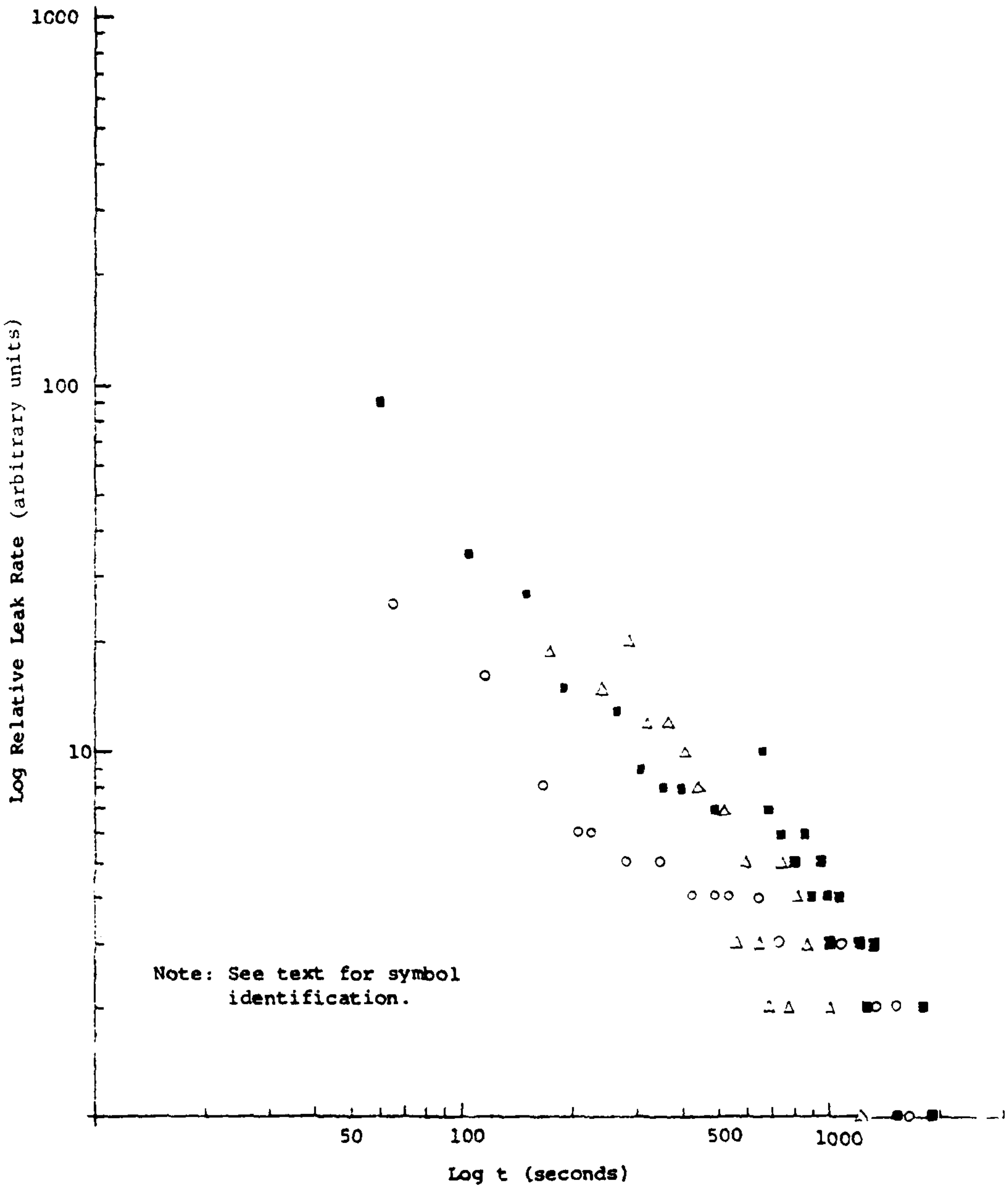

Figure 13. Leak rates of live detonators

APPENDIX

HELIUM LEAK TESTS

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I. TEST RESULTS

Two types of tests were performed on the detonators. The first was a standard "bombing" test. (This test gets its name from the pressure vessel or "bomb" in which the samples are pressurized with helium prior to testing.) Samples from each package of detonators were bombed and tested. Three runs were made, all at 4 atmospheres (absolute) of helium pressure. The bombing times for the three runs were 1 hour, 2 hours and 3 hours.

The most coherent test results were achieved with the detonators drilled with .001" diameter holes. These are presented in graph form in Figure A-1, which shows the measured leak rate as a function of time-before-test (this is measured from the time the helium pressure in the bomb is released and the helium flushed out). Because the data is plotted in logarithmetic form, the exponential signal decay should be apparent as a straight line.

The data on the graph make clear that regardless of bombing time, the signal from all pieces was within a single (though somewhat broad) envelope. All lines connect data points generated by retesting a detonator at intervals after removing it from the bomb. The tests after 1 hour of bombing show as many as three tests per piece; after the 2 hour bombing, there were no retests made; and after the 3 hour bombing, a few of the pieces were retested.

Differences in the slope of the straight lines represent, mathematically, differences in the exponents of "e" in the equation

$$S_t = P \left[1 - e^{-3600 \frac{L}{V} T} \right] \left[e^{-\frac{L}{V} t} \right] \quad L \qquad (1)$$

which is discussed in the appended paper, "Helium Mass Spectrometer Leak Testing of Pressure-"Bombed" Sealed Enclosures". Since T (the bombing time) does not change from one detonator to the next within a batch; and

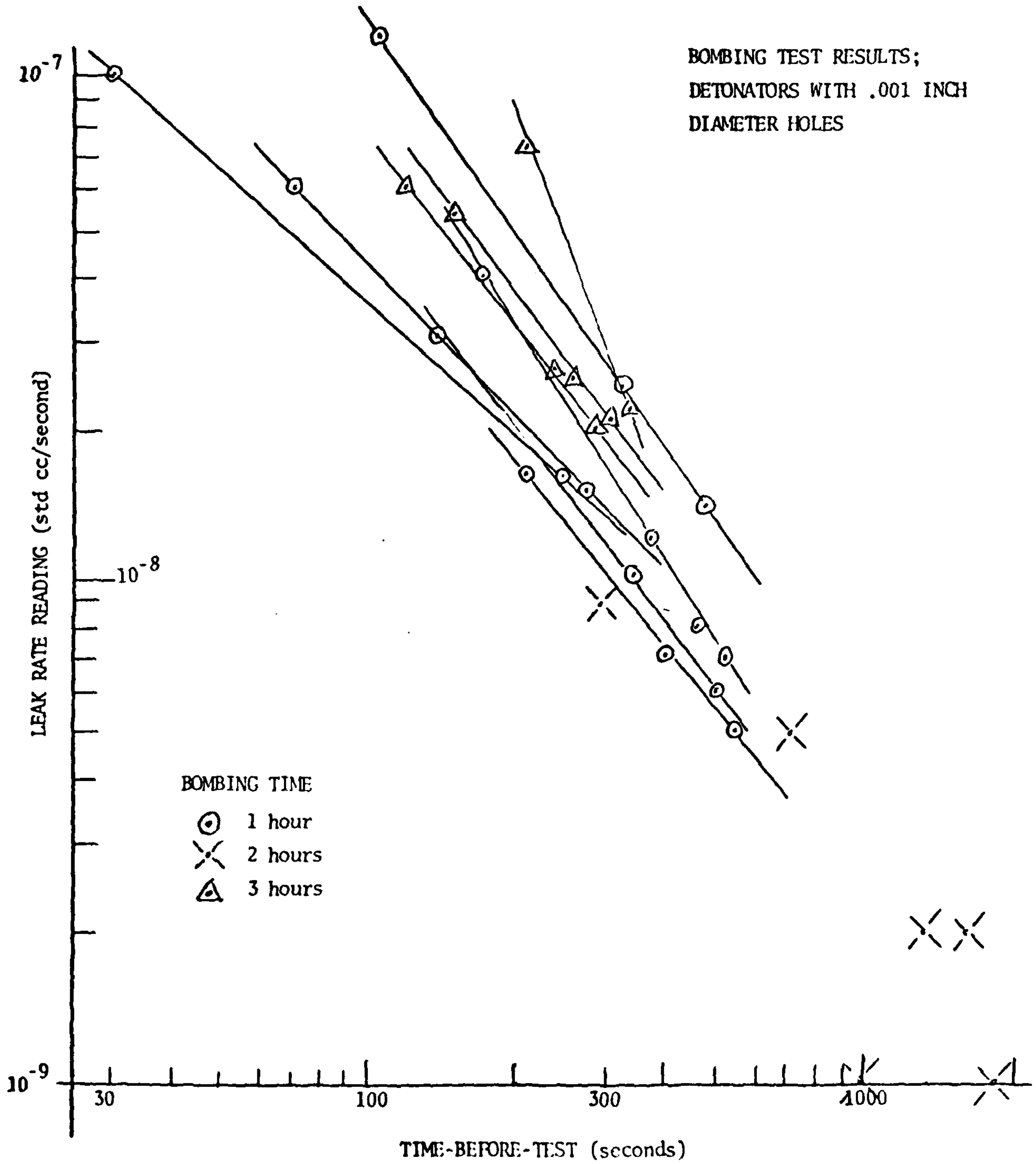
BOMBING TEST RESULTS;
DETONATORS WITH .001 INCH
DIAMETER HOLES
LEAK RATE READING (std cc/second)
10^{-7}
10^{-8}
10^{-9}
30
100
300
1000
BOMBING TIME
1 hour
2 hours
3 hours
TIME-BEFORE-TEST (seconds)

Figure A-1

t (the wait time) is a parameter of the experiment, the slope of the line is, directly, $\frac{L}{V}$ or the ratio of the leak rate to the volume. At first glance it seems clear that all parts have the same volume; and that a faster decay (or steeper curve) indicates a larger leak (i.e., a .001 diameter hole without a burr, or somehow drilled oversize). Another possibility is mentioned below.

The fact that the data for all tests lie in the same band simply demonstrates that the part is filled with helium before even the 1 hour bombing time is completed, and that further bombing does not introduce more helium into the part.

The actual conductance through the .001 diameter holes was measured and found to be about 0.4 std. cc/sec. This was measured for several detonators by opening, emptying, then sealing them into the wall of a vacuum system. The pressure (P) in the system resulting from the air flow through the .001" diameter hole was measured and multiplied by the speed (S) of the vacuum pumps to determine the conductance (Q)

$$Q = PS.$$

Measurements were also made with the detonator opened but the load undisturbed. This method measured the conductance through the load to the far wall of the container. The flow rate was about 1.3×10^{-2} std. cc/sec. The effective leak rate for the package is somewhere between these limits; larger than 1.3×10^{-2} and smaller than 4×10^{-1} std. cc/sec. This is the value of L to be used in the equation 1. The volume of the detonator is about 3.5×10^{-2} cc, so L/V is somewhere between 0.3 and 10 -- a value higher than any considered in the paper referenced. Data from that paper, or use of the equation, suggest that the helium in the part would exit so quickly as to give a "zero" reading on a leak detector. The fact that it doesn't may be due to the detonator filling material, as it adsorbs the helium on its surface wither sinking a great volume of helium or simply delaying its desorbtion as a gas for some tens of seconds. Only such a mechanism would yield the leak detector measurements

which have been observed. Furthermore, any variations in the filling material, whether in the mass of it or some other physical characteristic (e.g. vapor content) might also explain the variations in rate of release of helium seen as different slopes on the graph in Figure A-1.

All measurements of detonators with larger holes are consistent with the results discussed above; that is, all tests showed signals decreasing with time-after-test, and all signals stronger than predicted by considerations of L/V. It is interesting to note that the signals from detonators with hole sizes from .001" diameter through .02" diameter exhibit very similar signal levels at the same early time-before-test, though the ones with larger holes show a somewhat faster decay rate. This is consistent with the concept of adsorbtion of helium on the filling material within the detonator.

The control detonators (with no holes drilled in the case) yielded various low leak rates, which did not decay with time (indicating small leaks), mixed with some high leak rates (indicating larger leaks) and some "zero"-level signals. There was no indication that the lacquer was adsorbing helium.

All of the drilled detonators exhibited large signals if tested within a few seconds of immersion in helium. This was true if the immersion was a simple atmospheric pressure bath for a minute, followed by immediate testing. (The testing itself is, of course, preceded by an evacuation phase which lasts about 10 seconds.) The fact that even the detonators with largest holes yielded a signal in this situation suggests that this would be an excellent gross leak test which could quickly identify gross leakers before a full bombing cycle was carried out.

SUMMARY

The test results discussed here show that (1) regardless of bombing time, tests for leaks of 10^{-1} std. cc/sec must be made with minimal wait times; (2) leaks of 10^{-1} std. cc/sec appear (with the use of the bombing tables) to be much smaller, in the 10^{-4} std. cc/sec range; and (3) that there are fairly wide differences between apparently identical detonators.

II. FURTHER TESTING

The first comment to be made in any case of "bomb" testing is that the test would be better (easier, faster and more reliable) if helium can be introduced into the package at the time it is filled and sealed. When this is done, true leak rates can be measured directly (assuming the leak rate is not so large as to empty the container before the test). This approach can do more for improving tests than any other suggestion.

It is a basic requirement of the design of leak testing to determine the largest acceptable leak. This can, of course, be a very difficult undertaking. Without a "reject specification", however, meaningful quality control cannot be achieved.

Finally, it may be reassuring to generate leaks at the reject level (perhaps by laser-drilling or electron beam drilling) in blank cups and test them at different bombing pressures and times after filling with live materials (to avoid incomplete simulation of the real product). These tests will provide the information necessary to design appropriate test equipment.

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HELIUM LEAK TESTING: A NEW ANALYSIS

by

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and

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HELIUM LEAK TESTING:
a new analysis

by Walton E. Briggs and Sherwood G. Burnett

Wherein the testing of sealed electronic circuits is investigated with the aid of special Tables designed for working with a remarkable tool: The Helium Mass Spectrometer.

To insure long life and reliability, small electronic devices, such as transistors, diodes, and integrated circuits, must be sealed against hostile environments. Earthbound devices can suffer from moisture penetration, salt spray and airborne contaminants. In outer-space, loss of gas from the enclosure can cause failure from reduced cooling and from subtle chemical changes in the substrate itself.

Devices and integrated circuits are sealed in packages of various types, such as the TO series, flatpacks, dual in-line packs (DIPs), etc., containing the requisite gaseous environment and providing leads to the outside. Since no method has yet been developed to eliminate the possibility of leaks, tests must be performed to detect and eliminate leaking packages. Typically, the incidence of such faulty packages is 1-2%.

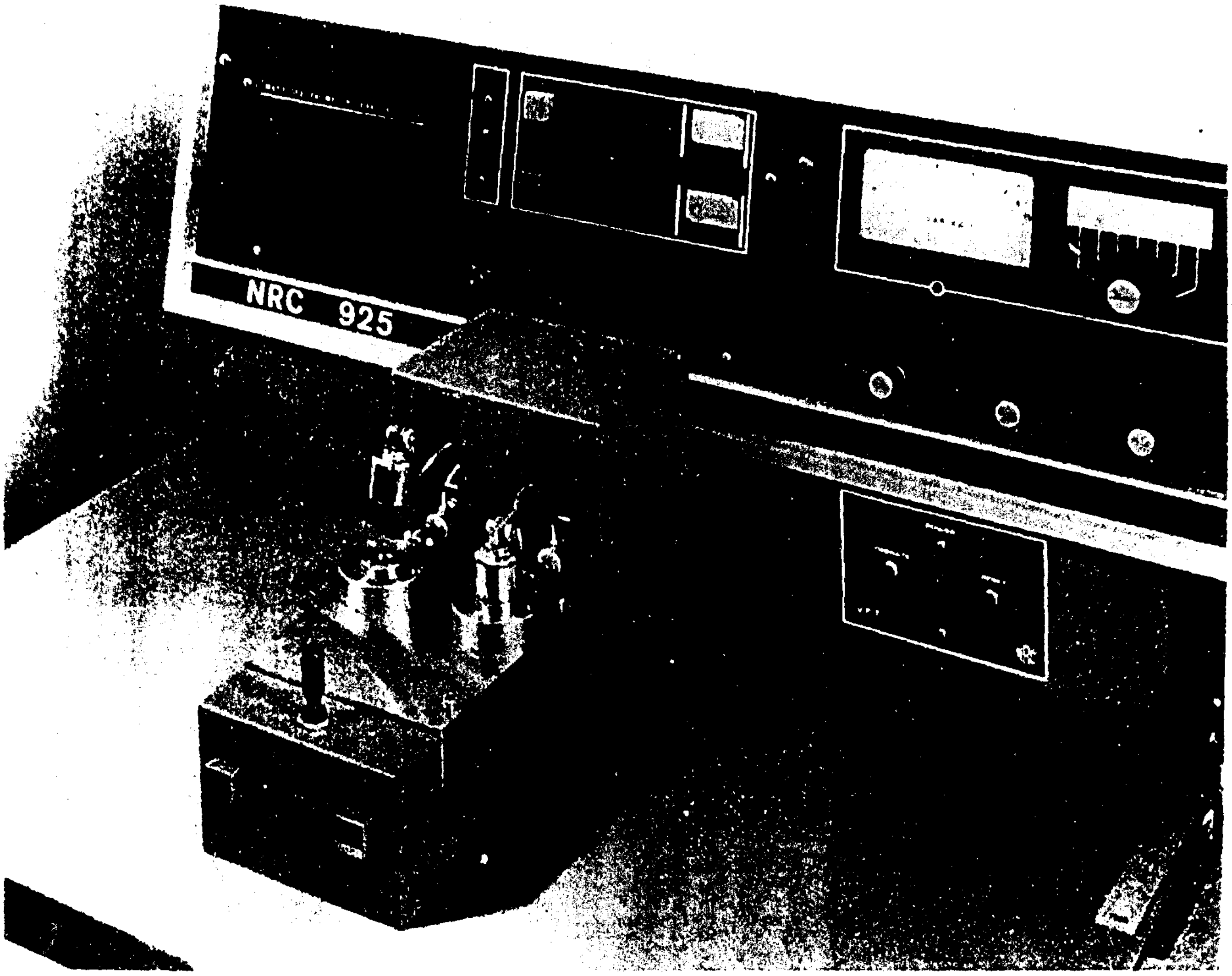
Helium Leak Detection

The most commonly used system for detecting leaks is the helium mass spectrometer. Helium is the lightest inert gas, its molecules will penetrate, though not close, the smallest leak. In addition, it is non-hazardous, reasonable in cost, available and present only in trace form in the atmosphere (about one part in 200,000). The equipment is reliable, easy to service, low in cost and it provides unmistakable indication of the presence of leaks. Since a mass spectrometer operates in vacuum, a vacuum-pumping system is incorporated, together with a "rough" pump to evacuate the test chamber, and a valving system to transfer the chamber to the spectrometer.

Testing of "Headers"

Two types of testing are carried out: 1) on lead-through assemblies (prior to installation of circuit); 2) on completed, sealed packages. The lead-through may be in the form of a TO header or an open flat pack, etc. Each metal-to-ceramic or metal-to-glass seal around a lead constitutes a potential leak, so testing at this stage can save some assembly costs.

One side of the unsealed lead-through assembly or "header" is temporarily sealed to the vacuum system of the leak detector with an O-ring, or other suit-



The Helium Mass Spectrometer. In use, part to be tested is placed in the left or right test port, and selector switch moved to that side. Plunger valve moves forward, connecting test port to two sequential stages of rough pumping and then to leak detector vacuum system, where helium presence is determined.

able sealing medium. Helium is applied to the other side at the moment of test, displacing the air in the vicinity of the test piece. Helium entering the leak detector indicates the presence and size of a leak. Concentration is essentially 100%, so the indication on the leak detector is the actual size of the leak.

In a few sealed packages, sealed-in helium permits leak testing by placing the device in a test enclosure connected to the vacuum system of the leak detector. Helium escaping from any leak is picked up by the mass spectrometer leak detector. Helium atmospheres in packaged electronic devices, although highly desirable from the testing viewpoint, are not common. In fact, the overwhelming majority of packages are sealed without helium and must therefore be "bombed" with helium for testing. This is done by placing them in a container, which is then pressurized to several atmospheres of helium for several hours. If a package leaks, helium enters the enclosure to be picked up later by the mass spectrometer leak detector just as though the package had originally contained helium.

Theory of Bombing

An impressive aspect of the "bombing" technique is its reliability and predictability. This despite a significant number of variables which can affect the amount of helium present within the enclosure after bombing, and, consequently, the relationship between the indicated leak and the actual leak. Some of these variables are internal volume, bombing time and pressure, and waiting time before testing. It is the purpose of this article to show why helium leak detection of "bombed" packages is reliable and therefore in widespread use today. The two factors which contribute most to reliability are:

- The strong evidence that very few leaks exist which are smaller than 5×10^{-7} std cc/sec.
- The surprisingly close relationship between the indicated leak and the actual leak in bombed packages.

This latter point is very important; the bombing technique has been used for several years, yet confusion still exists in the interpretation of test results. To develop a better understanding of the scope (and limitations) of this method, a series of quick-reference tables has been prepared, using a formula derived from the basic gas laws. These laws govern both in-flow of helium during bombing and out-flow after bombing and prior to testing. (They apply to any gases passing through the leak, but the only gas of particular interest in leak detection is the trace gas.)

The formula is:

$$S_t = [1 - e^{-3600aT}][e^{-at}]L$$

where:

S_t = indicated leak in std cc/sec.

T = bombing time in hours (the 3600 converts to seconds).

t = waiting time in seconds (interval between bombing and testing).

L = actual leak in std cc/sec/per atmosphere absolute.

$$a = \frac{L}{V} = \frac{\text{actual leak in std cc/sec*}}{\text{volume in cc}}$$

The formula is similar to that for charging and discharging a capacitor. The first bracket is the concentration of helium in the test piece after T hours of bombing. This may be anywhere from a small fraction of 1% up to 500% when bombing at five atmospheres absolute (approximately 60 psi gauge). The second bracket is the percent of that concentration left after waiting t seconds before testing. The first two terms, therefore, give the final concentration at the time of test in terms of percent of the original volume.

*The formula is based on one atmosphere absolute of helium. However, typical bombing pressure is five atmospheres absolute. Since flow rate through a leak is at least a linear function of pressure, multiply S_t by 5 to obtain the correct value for five atmospheres.

TABLE I: VOLUME 10^{-3} cc

(all leak rates in std cc/sec)

Actual Leak Rate (L)	Bombing Time (T)	WAIT TIME (t) 300 sec	1,000 sec	3,000 sec	10,000 sec
1x10(-5)	1 hr.	2.5x10(-6)	2.3x10(9)	[illegible]	[illegible]
	3 hrs	2.5x10(-6)	2.3x10(9)	[illegible]	[illegible]
	10 hrs	2.5x10(-6)	2.3x10(9)	[illegible]	[illegible]
1x10(-6)	1 hr.	3.6x10(-6)	1.8x10(-6)	2.4x10(-7)	2.2x10(-10)
	3 hrs	3.7x10(-6)	1.8x10(-6)	2.5x10(-7)	2.3x10(-10)
	10 hrs	3.7x10(-6)	1.8x10(-6)	2.5x10(-7)	2.3x10(-10)
1x10(7)	1 hr	1.5x10(-7)	1.4x10(-7)	1.1x10(-7)	5.6x10(-8)
	3 hrs.	3.2x10(-7)	3.0x10(-7)	2.4x10(-7)	1.2x10(-7)
	10 hrs	4.7x10(-7)	4.4x10(-7)	3.6x10(-7)	1.8x10(-7)
1x10(-8)	1 hr	1.8x10(-9)	1.8x10(-9)	1.7x10(-9)	1.6x10(-9)
	3 hrs.	5.1x10(-9)	5.1x10(-9)	5.0x10(-9)	4.6x10(-9)
	10 hrs.	1.5x10(-8)	1.5x10(-8)	1.5x10(-8)	1.4x10(-8)

TABLE II: VOLUME 10^{-2} cc

(all leak rates in std cc/sec)

Actual Leak Rate (L)	Bombing Time (T)	300 sec	1,000 sec	3,000 sec	10,000 sec
1x10(5)	1 hr	3.6x10(-5)	1.8x10(5)	2.4x10(6)	2.2x10(9)
	3 hrs	3.7x10(-5)	1.8x10(5)	2.5x10(6)	2.3x10(9)
	10 hrs	3.7x10(-5)	1.8x10(5)	2.5x10(6)	2.3x10(9)
1x10(6)	1 hr	1.5x10(-6)	1.4x10(6)	1.1x10(6)	5.6x10(7)
	3 hrs	3.2x10(-6)	3.0x10(6)	2.4x10(6)	1.2x10(6)
	10 hrs	4.7x10(-6)	4.4x10(-6)	3.6x10(-6)	1.8x10(6)
1x10(-7)	1 hr	1.8x10(-8)	1.8x10(-8)	1.7x10(8)	1.6x10(8)
	3 hrs	5.1x10(-8)	5.1x10(-8)	5.0x10(8)	4.6x10(8)
	10 hrs	1.5x10(-7)	1.5x10(-7)	1.5x10(7)	1.4x10(7)
1x10(-8)	1 hr	1.8x10(-10)	1.8x10(10)	1.8x10(10)	1.8x10(10)
	3 hrs	5.4x10(-10)	5.4x10(10)	5.4x10(-10)	5.3x10(10)
	10 hrs	1.8x10(-9)	1.8x10(9)	1.8x10(9)	1.8x10(9)

TABLE III: VOLUME 10^{-1} cc

(all leak rates in std cc/sec)

Actual Leak Rate (L)	Bombing Time (T)	WAIT TIME (t) 300 sec	1,000 sec	3,000 sec	10,000 sec
1x10(-5)	1 hr	1.5x10(-5)	1.4x10(5)	1.1x10(5)	5.6x10(6)
	3 hrs	3.2x10(-5)	3.0x10(-5)	2.4x10(5)	1.2x10(5)
	10 hrs	4.7x10(-5)	4.4x10(5)	3.6x10(5)	1.8x10(5)
1x10(6)	1 hr	1.8x10(-7)	1.8x10(7)	1.7x10(7)	1.6x10(7)
	3 hrs	5.1x10(-7)	5.1x10(7)	5.0x10(7)	4.6x10(7)
	10 hrs	1.5x10(6)	1.5x10(6)	1.5x10(6)	1.4x10(6)
1x10(7)	1 hr	1.8x10(9)	1.8x10(9)	1.8x10(9)	1.8x10(9)
	3 hrs	5.4x10(9)	5.4x10(9)	5.4x10(9)	5.3x10(9)
	10 hrs	1.8x10(8)	1.8x10(8)	1.8x10(8)	1.8x10(8)
1x10(8)	1 hr	1.8x10(-11)	1.8x10(11)	1.8x10(11)	1.8x10(-11)
	3 hrs	5.4x10(-11)	5.4x10(-11)	5.4x10(-11)	5.4x10(-11)
	10 hrs	1.8x10(-10)	1.8x10(-10)	1.8x10(-10)	1.8x10(-10)

Example	Description	Ref. Table	Volume (V)	Actual Leak Rate (L) std cc/sec
1	Small Volume and Large Leak	I	10(-3) cc	1 x 10(-5)
2	Small Volume and Small Leak	I	10(-3) cc	1 x 10(-8)
3	Large Volume and Large Leak	IV	1 cc	1 x 10(-5)
4	Large Volume and Small Leak	IV	1 cc	1 x 10(-8)

TABLE IV. VOLUME 1 cc

(all leak rates in std cc sec)

[illegible]	[illegible]	[illegible] 6	1 8x10(6	1 7x10(6)	1 6x10(6)
	[illegible]	[illegible] 6	5 1x10(6	5 0x10(6)	4 6x10(6)
	[illegible]	[illegible] 5	1 5x10(5)	1 5x10(5)	1 4x10(5)
	1 hr	1 8x10(8)	1 8x10(8)	1 8x10(8)	1 8x10(8)
1x10(6)	3 hrs	5 4x10(8)	5 4x10(8	5 4x10(8)	5 4x10(8)
	10 hrs	1 8x10(7)	1 8x10(7)	1 8x10(7)	1 8x10(7)
	1 hr	1 8x10(10)	1 8x10(10)	1 8x10(10)	1 8x10(10)
1x10(7)	3 hrs	5 4x10(-10)	5 4x10(-10)	5 4x10(-10)	5 4x10(-10)
	10 hrs	1 8x10(9)	1 8x10(9)	1 8x10(- 9)	1 8x10(- 9)
	1 hr	1.8x10(—12)	[illegible]	[illegible]	[illegible]
1x10(8)	3 hrs	5.4x10(—12)	[illegible]	[illegible]	[illegible]
	10 hrs	1.8x10(—11)	[illegible]	[illegible]	[illegible]

TABLE VI: ABBREVIATED FORM

VOLUME: 10^{-3} cc

Actual Leak Rate (L)	Bombing Time (T)	WAIT TIME (t) 300 sec	1,000 sec	3,000 sec	10,000 sec
	1 hr	2 5x10(- 6)	2 3x10(- 9)	[illegible]	[illegible]
1x10(-5)	3 hrs	2 5x10(- 6)	2 2x10(- 9)	[illegible]	[illegible]
	10 hrs	2 5x10(- 6)	2 3x10(- 9)	[illegible]	[illegible]
	1 hr	3 6x10(- 6)	1 8x10(- 6)	2.4x10(- 7)	2 2x10(-10)
1x10(-6)	3 hrs	3 7x10(- 6)	1 8x10(- 6)	2 5x10(- 7)	2 3x10(-10)
	10 hrs	3 7x10(- 6)	1 8x10(- 6)	2 5x10(- 7)	2 3x10(-10)
	1 hr	1 5x10(- 7)	1 4x10(- 7)	1 1x10(- 7)	5 6x10(- 8)
1x10(-7)	3 hrs	3 2x10(- 7)	3 0x10(- 7)	2 4x10(- 7)	1 2x10(- 7)
	10 hrs	4 7x10(- 7)	4 4x10(- 7)	3 6x10(- 7)	1 8x10(- 7)

VOLUME: 10^{-2} cc

Actual Leak Rate (L)	Bombing Time (T)	300 sec	1,000 sec	3,000 sec	10,000 sec
	1 hr	3.6x10(- 5)	1.8x10(- 5)	2 4x10(- 6)	2.2x10(- 9)
1x10(-5)	3 hrs	3 7x10(- 5)	1.8x10(- 5)	2.5x10(- 6)	2.3x10(- 9)
	10 hrs	3 7x10(- 5)	1 8x10(- 5)	2.5x10(- 6)	2.3x10(- 9)
	1 hr	1 5x10(- 6)	1 4x10(- 6)	1 1x10(- 6)	5 6x10(- 7)
1x10(-6)	3 hrs	3 2x10(- 6)	3.0x10(- 6)	2.4x10(- 6)	1 2x10(- 6)
	10 hrs	4 7x10(- 6)	4.4x10(- 6)	3 6x10(- 6)	1.8x10(- 6)
	1 hr	1 8x10(- 8)	1 8x10(- 8)	1.7x10(- 8)	1.6x10(- 8)
1x10(-7)	3 hrs	5.1x10(- 8)	5.1x10(- 8)	5 0x10(- 8)	4 6x10(- 8)
	10 hrs	1 5x10(- 7)	1.5x10(- 7)	1 5x10(- 7)	1.4x10(- 7)

These tables each represent a series of values for the indicated leak S_t for test pieces having internal volumes of 10^{-3}, 10^{-2}, 10^{-1}, and 1 cubic centimeter, respectively. Table 5 is made up from Tables 1 and 4 and is graphed in Fig. 1 & 2. Table 6 is an abbreviated form for realistic detection of integrated circuit packages whose volumes are seldom more than 10^{-2} cc.

TABLE V

$\frac{L}{V}$	Indicated Leak Rate S (std cc/sec) at 300 sec	at 1000 sec	at 3000 sec	DETECTABILITY
10(−2)	2 3 x 10(−6)	2.3 x 10(− 9)	4 7 x 10(−18)	GROSS LEAK (detectable within 1000 sec)
10(−5)	5.1 x 10(− 9)	5 1 x 10(− 9)	5 0 x 10(− 9)	FINE LEAK (detectable)
10(−5)	5.1 x 10(− 6)	5 1 x 10(− 6)	5 0 x 10(−6)	FINE LEAK (detectable)
10(−8)	5 4 x 10(−12)	5 4 x 10(−12)	5 4 x 10(−12)	MARGINAL (increase P or T)

For example, if bombing results in 80% concentration and the wait time depletes this to 90% of its original value, we have

$S_t = (80\%)(90\%)L = 72\% L.$

If the actual leak, L, is 5 x 10[illegible] std cc/sec, the indicated leak, S_t, will be 3.6 x 10[illegible] std cc/sec.

Leak Rate Tables

Four tables have been prepared (see boxes left), each representing a series of values for the indicated leak S_t for test pieces having internal volumes of 10^{-3}, 10^{-2}, 10^{-1}, and 1 cubic centimeter, respectively. Each table encompasses a range of four actual leak rates, three bombing times and four wait times, giving a total of 48 values of S_t. All figures are calculated for a bombing pressure of five atmospheres absolute. These tables are very useful to determine whether or not a detectable signal can be expected for a given test object and, if not, whether increased bombing time and/or pressure and/or shorter wait time will help.

For example:

- TO5 with net internal volume of 10^{-2} cc (Table 2) and an actual leak of 1 x 10 [illegible] std cc/sec bombed for three hours at five atmospheres of helium absolute (about 60 psig).
  - S_t after 300 sec (5 min) → 5.1 x 10 [illegible] std cc/sec
  - S_t after 10,000 sec (2¾ hrs.) → 4.6 x 10 [illegible] std cc/sec

The concentration of helium in the TO5 at the time of test is 51% and 46% respectively. Note that the decrease is small.

- A TO5 with identical parameters except with an actual leak of 1x 10 [illegible] std cc/sec:
  - S_t after 5 min. → 3.2 x 10 [illegible] std cc/sec
  - S_t after 2¾ hrs. → 1.2 x 10 [illegible] std cc/sec

The concentration of helium in the TO5 at the time of test is 320% and 120%, respectively, (3.2 and 1.2 atmospheres absolute). Note the more rapid decrease in this example.

Test Categories

A glance at the tables will reveal that the majority of indicated leak rates are detectable; the few that are not are shaded. Three categories can be defined:

"Fine" Leakers: Those detectable by the leak detector (the majority of figures in all four tables).

"Gross" Leakers: Those whose loss of helium is so rapid that no detectable signal is present at the time of test. (shaded area in Table 1 if test is not carried out within 1000 seconds or 17 minutes).

Marginal: Those which can be

tested by increase in bombing pressure or time (shaded areas in Tables 3 and 4).

To illustrate these categories, consider four different examples (Fig. 1-2) obtained by using the smallest and largest volumes and the largest and smallest leak rates. For uniformity of comparison, assume three hours bombing time. From Tables 1 and 4, then, we can make up Table 5 showing these four examples.

Referring to this new table, notice that in Example 1 (Fig. 1), the decay in helium concentration is quite rapid in this gross leaker, but if testing is done within about 17 minutes, the leak can be detected. It is interesting to go back to Table 1 at this point. Note that bombing *time* has no effect on helium concentration after 1 hour, because the cavity is already full of helium (five atmospheres). Bombing *pressure*, however, if increased *would* increase the indicated leak rate.

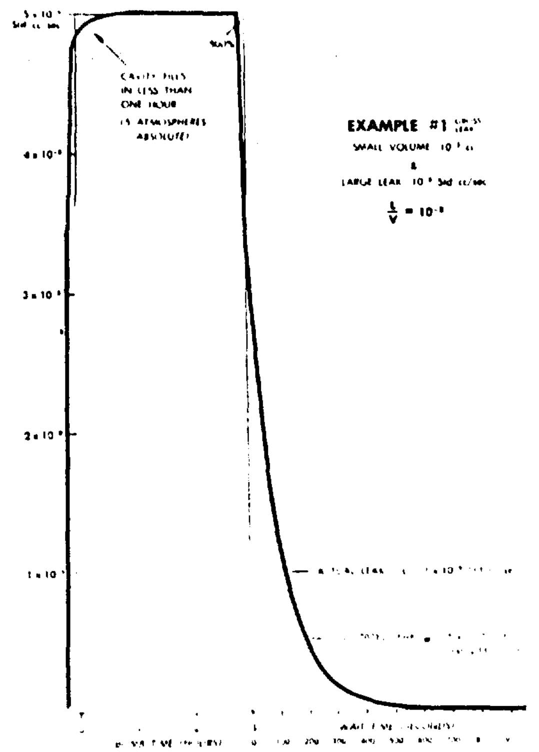
In Example 4 (Fig. 2), a reference back to Table 4 shows that extended bombing *time* can bring the indicated leak into the detectable range. Note that the indicated leak increases linearly with bombing time (because the leak is so small and the volume so large). One can extrapolate to determine that the indicated leak would be about 5.4 x 10^{-10} std cc/sec after 30 hours of bombing, and so on.

The gross leak can be detected within about 17 minutes. The marginal leak can be detected by increasing bombing time and/or pressure. Thus, the tables can predict the conditions under which leaks in your product can be detected.

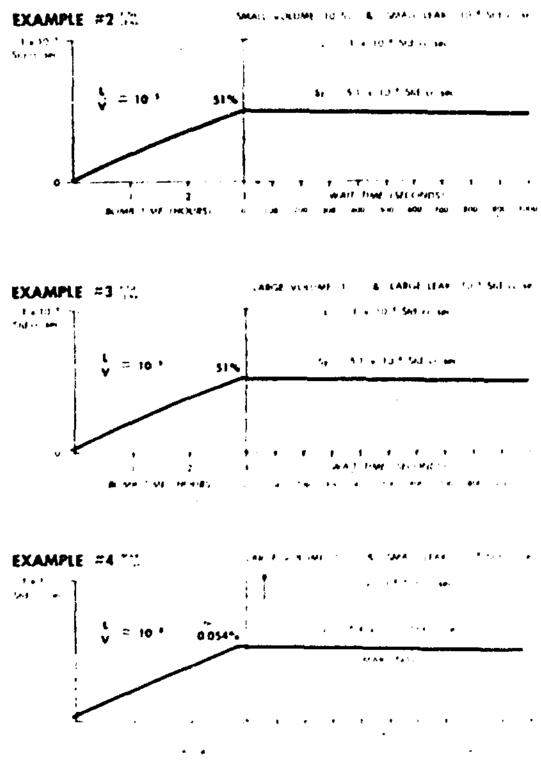
In example 1 (gross leak), note that the cavity is already filled in less than one hour. Thus, extended bombing has no effect upon the ultimate signal. Once it is removed from the bomb, the rate of loss is rapid. The cross lines show that the nominal value of the leak will be reached in about three minutes and 50% of the value of the leak will be reached in about four minutes.

In the other (Fig. 2) three examples, the rate of rise is nearly linear because the cavity does not approach the condition of being filled with five atmospheres, or 500% of helium. In examples 2 and 3, in fact, it approaches only one-tenth of this value or 50% of one atmosphere absolute. Since the rate of loss is very low, it is possible to test two or three hours after bombing without significant change in the indicated leak, which, in this case, happens to be about 50% of the actual value of the leak.

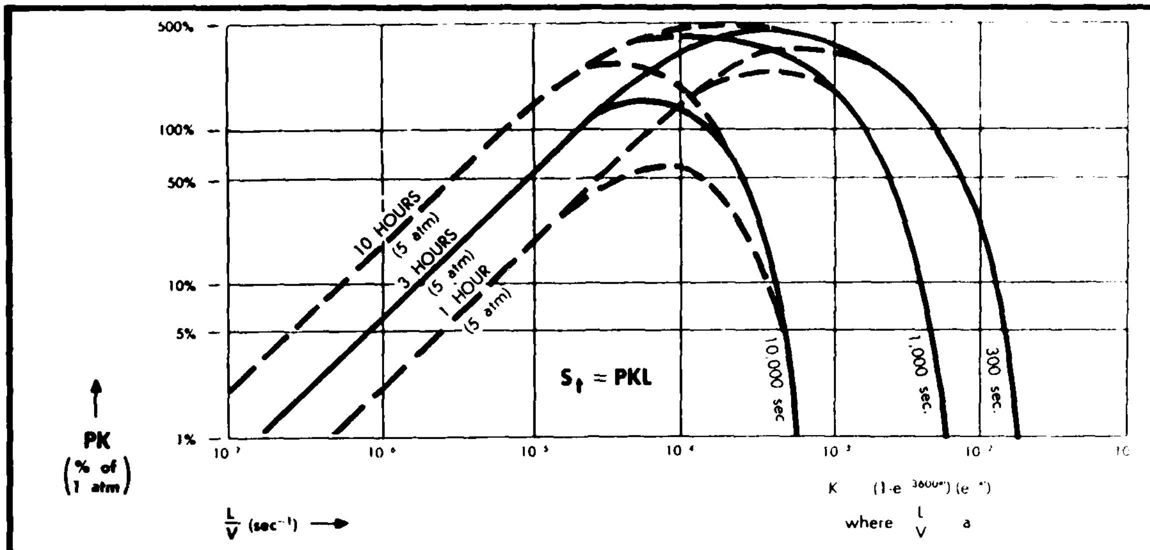
With reference to the rapid loss of helium from the gross leaker in example 1, it is important to note that this loss is just as great whether the environment is atmosphere or vacuum. It is a common misconseption that the helium loss is more rapid during the rough pumping portion of the leak cycle. This is not true; the loss is just as rapid at atmospheric pressure, because the partial pressure of helium in the atmosphere is extremely low (about four microns in ordinary air). To the helium molecules inside the cavity escaping through the leak, the outside environment



1. Graphic example of gross leak—loss of helium is so rapid that no detectable signal is present at the time of test (shaded area, Table 1).



2. Examples of fine leak—detectable by leak detector; and marginal leak—testable by increases in 'bombing' pressure or time (shaded area, Tables 3 & 4)



3. **Plot showing per cent of trace gas in an enclosure vs. internal volume for a bomb time of three hours at five atmospheres pressure** *and wait times of 300, 1000 and 10,000 seconds.*

has essentially no helium and it therefore offers essentially no more back diffusion of helium than a vacuum environment.

In studying these curves, one observes that the ratio of the actual leak to the internal volume, or L/V, is an important parameter in determining the nature of the in-flow and out-flow of trace gas for a given bombing and waiting time. Note that in examples 2 and 3, L/V is the same. As a result, so is the percent of trace gas in the enclosure, namely, 51%. Thus, the value of S_t is just 51% of L.

Shown in Fig. 3 is a plot of the percent of trace gas in the enclosure, versus L/V, for bomb time of three hours at five atmospheres pressure and wait time of 300, 1000, and 10,000 seconds. Similar curves can be drawn for other bomb times. (The percent, or K factor, is the double exponential bracket in the formula at the beginning of this article, here multiplied by five to take into account actual bomb pressure. The maximum possible value is 500% or five atmospheres in this case).

The use of this curve can be illustrated by referring again to example 2 in Fig. 2, where $L/V = 10^{-5}$ sec^{-1} ($L = 10^{-8}$ std cc/sec, $V = 10^{-3}$ cc). Following the 10^{-5} line up, note that it intersects the curve at 51%. Therefore, the indicated leak S_t is 51% of 1×10^{-8} or 5.1×10^{-9} std cc/sec. Note also that this is independent of wait time up to at least 10,000 seconds.

This percentage would apply also to any other test object bombed under these conditions, provided it has an L/V ratio of 10^{-5} sec^{-1}. For example, it would apply to a volume of 10^{-2} cc and a leak rate of 10^{-7} std cc/sec. Here, the indicated leak would be 51% of 1×10^{-7} or 5.1×10^{-8} std cc.

It also applies to example 3 in Fig. 2 where $L = 10^{-5}$ std cc/sec and $V = 1$ cc. Again, $L/V = 10^{-5}$ sec^{-1}, so 51% of 1×10^{-5} is 5.1×10^{-6} std cc/sec.

Even more interesting is example 1 (Fig. 1). Here the leak rate is 1×10^{-5} std cc/sec and the volume is 10^{-3} cc, so $L/V = 10^{-2}$ sec^{-1}. This intersects the 300-second curve only, at about 20%. Thus, the indicated leak will be 20% of 1×10^{-5} or 2×10^{-6} std cc if testing is performed 300 seconds (five minutes) after bombing is complete. Loss of trace gas is rapid, however. Although not shown on this graph, the 1000-second curve (17 minutes) intersects the 10^{-5} sec^{-1} line at .02%, giving a leak of 2×10^{-9} std cc/sec.

In a more general sense, this curve also shows the range of values of L/V in which the indicated leak S_t will be at least 1% of the indicated leak L. For the 10,000-second wait curve the range is from 2×10^{-7} sec^{-1} to 6×10^{-4} sec^{-1}. This can be translated in terms of actual leak rate L for a given volume. For example, for a volume of 10^{-2} cc, the range becomes 2×10^{-9} std cc/sec to 6×10^{-6} std cc/sec. For a volume of 10^{-3} cc, it becomes 2×10^{-10} std cc/sec to 6×10^{-7} std cc/sec. Thus this curve can predict the tie between S_t and L over values of the latter.

There is considerable evidence that few, if any, leaks in sealed electronic packages are smaller than 5×10^{-7} std cc/sec. Most are 10^{-6} std cc/sec or larger, caused by glass failing to wet metal or by cracks developing in glass subsequent to the sealing operation. It can be shown that the difference in coefficient of expansion will produce a leak of the order of 10^{-6} std cc/sec or larger. (In the TO series packages a poor or discontinuous weld will create a leak of even larger size.) If one accepts this thesis, certain interesting conclusions will follow. Referring again to Tables 1 through 4, the figures corresponding to 10^{-8} std cc/sec actual leak rate can be deleted. Then, further, if we consider that very few integrated circuit packages contain more than 10^{-2} cc internal volume, the tables become reduced to the abbreviated form shown in Table 6. Referring to this:

• Except for the gross leak in the first row, there is a remarkably close relationship between indicated leak rate and actual leak rate, generally within a factor of five (for bombing times of three hours or more).

• No marginal leakers are present. All give detectable signals without extremes of bombing or time.

Operators who are accustomed to set the reject point with considerable precision have noted that when a leak occurs, the leak rate meter usually pegs full scale. That is to say, very few leaks occur whose value is quite close to the reject point. Table 6 bears this out. A typical reject point is 1×10^{-8} std cc/sec. Leaving out the gross leaker (row number 1) and assuming that the test is carried out within 50 minutes (3000 seconds), most of the values of indicated leak would peg the meter.

Test Cycle—The Helium Mass Spectrometer

The test chamber contains air at atmospheric pressure when loaded with the test piece. Since the helium mass spectrometer leak detector operates with an internal vacuum, the test chamber pressure must be substantially evacuated before it can be connected to the leak detector's vacuum system. When headers are tested, they form part of the test chamber. In either case, initial evacuation is performed by a mechanical vacuum ("roughing") pump. Then the test port is disconnected from the roughing pump and connected to the leak detector's vacuum system by the use of valves.

This valving is automatic on modern mass spectrometer leak detectors, and the test time for one piece or one load of several pieces is approximately 30 seconds. Since 30 seconds per test is too long for testing large numbers of pieces individually in sequence, manifolds with as many as 25 individually valved ports have been used. If a leak is indicated, the leaking test piece can be isolated by manipulating the valves while watching the leak indicating meter. This method is moderately acceptable—if few leakers are present and if the individual valves are reliable. However, in actual practice, there is usually at least one leaker in each test, and skilled operators often have difficulty in completing a cycle in ten minutes. Moreover, errors in judgment and in valve manipulation may cause defective parts to be passed and good parts to be rejected.

The VFT Approach: To speed up this process and at the same time provide individual tests for each unit in sequence, an interesting new concept was developed called the VFT (Very Fast Test panel at right in equipment shown opposite title page). It gives positive leak test results with minimum operator skill and at nearly 10 times the speed of the multiport manifold method.

The VFT includes two pneumatic fast-acting plunger valves (activated by a selector switch), two test ports, two red leak indicator lamps and the associated electronic controls to operate it. In use, the part to be tested is placed in the left or right test port, and the selector switch is moved to that side. The plunger valve moves rapidly forward, connecting the test port to two sequential stages of rough pumping and then to the leak detector vacuum system, where the presence of helium is quantitatively determined. (In header testing, as seen opposite title page, helium is applied automatically to the outside during test) If the indicated leak rate exceeds a predetermined value, the plunger withdraws immediately, (isolating the test port from the leak detector), the red indicator lamp in front of the appropriate test port lights, and the VFT becomes inoperative. The operator rejects the defective part and pushes the lighted push button to reactivate the cycle. If the leak rate is not exceeded, the operator continues to unload and load whichever test port is not under test, moving the switch as needed.

Safety precautions built into the control circuitry prevent operation if the leak detector is not ready and prevent transfer of the test port to the leak detector if improperly loaded. A photocell monitors the master "test" lamp on the leak detector and a pressure switch in the second stage roughing line prevents the plunger valves from moving into the leak detector vacuum system if too high a pressure exists in the test port.

The plunger valves consist of ½-inch diameter chrome-plated cylinders sliding through four rubber O-rings and a dust shield. The O-rings isolate the air vent passage from the first rough pump, the first rough pump from the test port, the test port from the second rough pump, and the second rough pump from the leak detector. Two groups of radial openings in the plungers are connected axially so that, as the plunger moves, the test port is connected sequentially to the proper vacuum system (or is vented).

VFT Advantages

The plungers are actuated by air cylinders with adjustable valves to control the speed both in and out. If the test port volume is small, such as with a header, cycle time is less than three seconds.

The advantages of the VFT:

• Rejects are clearly defined and indicated.

• Operator error is eliminated by simple method.

• Helium saturation of the leak detector is eliminated (plunger withdraws instantly at a leak).

• Both detector and operator are fully occupied.

• Interchangeable port adapters, (see title page) provide maximum efficiency for various test pieces.

• Maintenance is very simple. The main wear occurs only on the plunger O-rings which are easily replaced since removable spacers are used instead of fixed grooves. O-ring life is in excess of 100,000 tests.

Finally, helium leak testing of sealed enclosures has come to be widely used and is considered to be reliable because:

1. Tests are definitive; test pieces are either tight or they leak in excess of 10^{-6} std cc/sec.

2. With few exceptions, bombed packages provide indicated leak rates comparable to the actual leak rate.

3. Fast test methods have been developed, such as the VFT, which combine high sequential speed with high reliability.

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